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WADC TECHNICAL REPORT 53-373

SUPPLEMENT 3

ASTIA DOCUMENT No. AD0110468

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**A REVIEW OF THE AIR FORCE  
MATERIALS RESEARCH AND  
DEVELOPMENT PROGRAM**

HELEN E. HINES

RUTH F. WALDEN

MATERIALS LABORATORY

BEST AVAILABLE COPY

OCTOBER 1956

Statement A  
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WRIGHT AIR DEVELOPMENT CENTER

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MATERIALS RESEARCH AND  
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*OCTOBER 1956*

**WRIGHT AIR DEVELOPMENT CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO**

## FOREWORD

This report was prepared by Miss Helen E. Hines and Mrs. Ruth F. Walden of the Technical Data Section, Technical Services Branch, Materials Laboratory, Directorate of Research, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

Technical Reports prepared by Materials Laboratory project engineers (those having no contract number) and Materials Laboratory contractors during the period 1 July 1955 - 30 June 1956 are abstracted herein.

Abstracts for Technical Notes of general public interest and Technical Reports not previously included are given in this supplement.

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## ABSTRACT

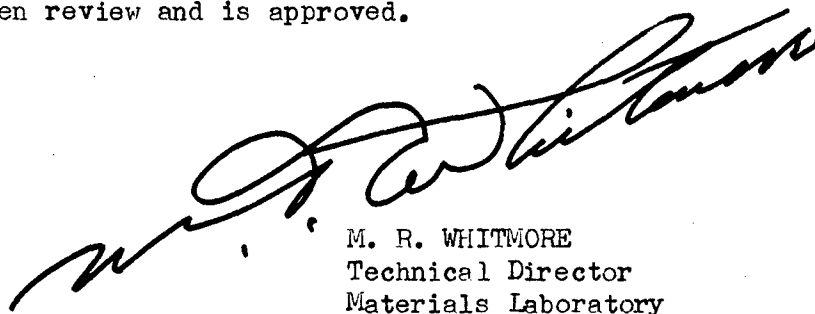
One hundred and forty-five (145) technical reports and technical notes written during the period 1 July 1955 - 30 June 1956 are abstracted. These reports cover the following areas of research: adhesives, metallurgy, analysis and measurement, biochemistry, textiles, petroleum products, plastics, packaging, protective treatments and rubber.

A contractor index, investigator index, and a numerical index of all the technical reports issued during the period March 1923 - June 1956 are provided.

## PUBLICATION REVIEW

This report has been review and is approved.

FOR THE COMMANDER:

A large, stylized handwritten signature in black ink, likely belonging to M. R. Whitmore, is written over the typed name and title.

M. R. WHITMORE  
Technical Director  
Materials Laboratory  
Directorate of Research

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ADHESIVES, STRUCTURAL

WADC TR 53-126 Part 1

March 1956

SUBJECT: ELEVATED TEMPERATURE RESISTANT MODIFIED EPOXIDE RESIN ADHESIVES FOR METALS

INVESTIGATOR: Marguerite Naps, Frank C. Hopper

CONTRACT: AF 33(616)-2430

CONTRACTOR: Shell Development Company

ABSTRACT: A heat-resistant adhesive, designated Adhesive 422, developed under a previous contract, was more thoroughly investigated. Adhesive 422 is based on a mixture of EPON 1001 and Plyophen 5023, filled with aluminum dust. Attempts to obtain improved performance by changing various ingredients or proportions in the basic formula, or by the addition of modifiers, were substantially unsuccessful. The surface preparation of the metal adherend and the use of primers were investigated. The properties of bonds to numerous adherends were determined.

More promising in resistance to aging at 500°F were new adhesives based on EPON X-15100.

WADC TR 54-98 Part 2

January 1956

SUBJECT: INVESTIGATION AND DEVELOPMENT OF HIGH TEMPERATURE STRUCTURAL ADHESIVES

INVESTIGATOR: Kenneth L. McHugh

CONTRACT: AF 33(616)-2448

CONTRACTOR: The Connecticut Hard Rubber Company

ABSTRACT: An epoxy modified silicone resin composition for use as a metal-to-metal adhesive has been developed which displays shear strength values slightly in excess of the target requirement of 1000 psi at 500°F, and shear strength values at room temperature of about 1100 psi. The highest shear strength value obtained at 500°F in an evaluation of seventy-seven commercial silicone and organo-modified silicone resins was 560 psi. A commercial organo-modified silicone resin produced a much higher shear strength at room temperature (average, 2300 psi), but a low shear strength at 500°F (average, less than 100 psi).

Experimental silicone resins were prepared which displayed shear strength values appreciably higher than those of the best commercial silicone resins. The best high-temperature shear strength values were found in resins having an R/Si ratio from 1.10 to 1.30 and a methyl content from 30 to 60 percent.

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Manuscript released by authors October 1956 for publication as a WADC Technical Report.

WADC TR 53-373 Sup 3

Epoxy-modified silicone resins were the most promising of almost one hundred modifications prepared with epoxy, alkyd, phenolic, and polyamine resins. Silicone-isocyanate copolymers, while not yet practical, showed interesting results. The Karl Fischer reagent was found useful for the determination of silanol groups in silicone resins.

Asbestine X and titanium dioxide fillers reinforced the silicone and organo-modified silicone resins to some extent. Glass fabric was of little or no value as a reinforcement.

WADC TR 55-87

April 1956

SUBJECT: TREATMENT OF METAL SURFACES FOR ADHESIVE  
BONDING

INVESTIGATOR: Samuel N. Muchnick

CONTRACT: AF 33(616)-2347

CONTRACTOR: The Franklin Institute Laboratories for  
Research and Development

ABSTRACT: A theoretical basis is presented for the adhesion of a metal to an organic bonding agent. The contact angle is shown to be an effective measure of the adequacy of a surface treatment for a metal surface. For example, when a water drop makes a low or a zero contact angle on an aluminum or a stainless steel surface immediately following a surface treatment, joint strength is generally at a maximum and most likely to be reproducible. A study has been made of the importance of the film absorbed on the metal surface and of the mechanical properties of the metal as they are related to nominal joint strength. Four different adhesives have been used to evaluate the surface treatments which result in the highest joint strengths. In addition, those procedural variables are investigated which might affect surface properties and joint strength.

WADC TR 55-271

March 1956

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT  
INORGANIC POLYMER STRUCTURAL ADHESIVES

INVESTIGATOR: Harold H. Levine

CONTRACT: AF 33(616)-2555

CONTRACTOR: Quantum, Inc.

ABSTRACT: Attempts to prepare inorganic metal-to-metal adhesives stable at 1000°F are described. The s-triazine and phosphonitrilic compounds were selected as the best approach because of their inherent thermal stability and reactivity.

Six promising products resulted from the following reactions:  
(1) melamine-phosphorus pentachloride (2) melamine-phosphorus oxychloride  
(3) melamine-phosphorus pentoxide (4) ammeline-phosphorus pentachloride (5)  
cyanuric trihydrazide-phosphorus pentachloride (6) carbohydrazide-phosphorus  
pentachloride. These products are stable from 750°F to red heat, are generally  
insoluble, and they possess very reactive atoms which may be capable of  
modifications resulting in adhesive properties.

WADC TR 53-373 Sup 3

Attempts to incorporate metal atoms into an adhesive precursor was successful with titanium.

Epoxy resins can be cured, without external heat, by use of phosphorus pentoxide.

Rigorous examination of the above six reactions is recommended. The effect of reaction conditions and reactant ratios, the degree of poly-functionality in starting materials, and the chemistry of the reactive atoms should be investigated to attain a desirable inorganic adhesive.

WADC TR 55-289

January 1956

SUBJECT: ROOM TEMPERATURE VULCANIZING SILICONE  
ADHESIVE  
INVESTIGATOR: Aldo J. DeFrancesco  
CONTRACT: AF 33(616)-2542  
CONTRACTOR: The Connecticut Hard Rubber Company  
ABSTRACT: All commercially available adhesives recommended for bonding silicone rubber to aluminum and to itself without the use of heat and pressure have been evaluated. Dow Corning A-4000, a two-component adhesive, fulfilled nearly all target requirements. Two catalysts, Dow Corning XY-22 and XY-27, were tested with A-4000; of the two, XY-27 is recommended. It is suggested that the 24-hour peel strength requirement be lowered from ten pounds per inch to eight pounds per inch, which would enable the A-4000:XY-27 combination to meet all the specifications of the contract.

A study of other room-temperature-curing systems with various modified siloxanes is reported. With the use of several curing systems which were developed during the course of the work, materials were prepared for bonding silicone rubber to aluminum and to itself. Poor bonds were obtained to aluminum. Good bonds were obtained to silicone rubber with the use of a cement compounded from Linde Y-1170, a hydrogenmethyl silicone fluid.

A literature survey of silicone adhesives and room-temperature curing systems is also included in this report.

WADC TR 56-24

March 1956

SUBJECT: PRELIMINARY INVESTIGATION OF LAMINATING  
TECHNIQUES FOR ALUMINUM SHEET MATERIAL  
INVESTIGATORS: Fred Werren  
B. G. Heebink  
CONTRACT: DO(33-616)53-20, Amend. A2(55-295)  
CONTRACTOR: U. S. Forest Products Laboratory  
ABSTRACT: The bonding of relatively large sheets of aluminum alloy to one another presents certain fabrication problems not experienced in the bonding of small areas. In an exploratory study of fabrication techniques, it was found that by varying the details of fabrication to suit the method of application and the specific adhesive, blister-free bonds of uniform quality could be made with all of the six commercial adhesives evaluated.

WADC TR 53-373 Sup 3

This report presents the results of preliminary investigations on the mechanical properties of 36- by 40-inch laminated panels consisting of 8 plies of 0.032-inch aluminum alloy. Five different adhesive systems were used to make the panels. Short-column edgewise-compression tests and flexural tests over short and long spans were run on the laminated material. The results show that, if the adhesive system used is rigid enough, the mechanical properties of the laminate, calculated on the basis of net area or net moment of inertia of the metal, are about equal to those of the same metal in solid form. If the shear modulus of the adhesive is low, buckling may occur at low loads under compressive edge loading, and excessive shear deflections may occur in flexure.

Some theoretical considerations concerning the behavior of laminated metal are presented in Appendix I.

#### ANALYSIS AND MEASUREMENT

WADC TR 53-201 Part 6

October 1955

SUBJECT: THERMAL CONDUCTIVITY AND HEAT CAPACITY OF  
MOLTEN MATERIALS

INVESTIGATORS: D. A. Ditmars  
D. C. Ginnings

CONTRACT: AF 33(616) 52-10

CONTRACTOR: National Bureau of Standards

ABSTRACT: The thermal conductivity of beryllium oxide has been measured from 40° to 750°C with an apparatus employing steady-state longitudinal heat flow along a rod of high-fired beryllium oxide surrounded by a "guard" tube with matching temperature gradient to minimize radial heat loss. The estimated accuracy of the measurements is about 2%. However, the values of thermal conductivity of the ideal BeO crystal are probably somewhat higher than the values given because of the low density (2.62 g/cm<sup>3</sup>) of the sample used.

WADC TR 54-268

February 1956

SUBJECT: QUANTITATIVE ANALYSIS OF ELASTOMERS THROUGH  
THE INFRARED SPECTRA OF THEIR PYROLYZATES

INVESTIGATOR: Freeman F. Bentley

ABSTRACT: A semi-quantitative method is described for determining polymer blends and co-polymer ratios from the infrared spectra of their pyrolyzates (distillates). The method was established through the analysis of phenolic resin blends in Buna N rubber, and by determining the percentage acrylonitrile in butadiene acrylonitrile co-polymers

The method described here permits the semi-quantitative analysis of Buna N phenolic resin blends, which because of their physical state, cannot be analyzed by conventional methods. It also makes it possible to estimate the amount of non-combustible materials in these polymers. The method is based on the pyrolyzate technique combined with standard infrared quantitative procedures.

WADC TR 53-373 Sup 3

November 1955

**SUBJECT:** INVESTIGATION OF THERMAL PROPERTIES OF  
PLASTIC LAMINATES, CORES, AND SANDWICH  
PANELS

**INVESTIGATOR:** F. R. O'Brien  
S. Oglesby, Jr.

**CONTRACT:** AF 33(616)-2045 S2 - (54-1233)

**CONTRACTOR:** Southern Research Institute

**ABSTRACT:** This report describes the measurement of the thermal properties of a selected group of plastic laminates, cores, and sandwich panels. Thermal conductivity, specific heat, and thermal expansion was determined for four glass-fabric laminates with the same reinforcing fabric but with different resins, four foam-plastic cores, one foam sandwich panel, and one honeycomb panel.

The equipment and procedures used in the thermal property measurements are described in WADC TR 54-306 Part I. The only change in procedure was the use of the method of least mean squares to define the enthalpy-temperature curves of the foam cores and the sandwich panels.

The thermal properties of the plastic laminates were in general agreement with those of the laminates tested in the first portion of this program. The foam cores, except the heat resistant core, decomposed at relatively low temperatures. Also, the low densities of these core materials gave less precision in the specific heat determinations.

WADC TR 55-421

February 1956

**SUBJECT:** COMPARATIVE METHODS OF FLUORINE ANALYSIS

**INVESTIGATOR:** Gordon D. Oshesky, 1/Lt. USAF

**ABSTRACT:** There are many methods for analyzing the fluorine content of fluoro-organic compounds. These methods involve varying degrees of convenience with regard to simplicity of apparatus, time required, and the skill of the operator and can be applied to a wide range of compounds. An attempt has been made in this project to determine which were best suited for compounds containing 10 to 70% fluorine. The methods discussed in this report include oxidation, reduction, gravimetric, and titrametric procedures. These methods are by no means all of the proposed methods, but a few of the more commonly used ones.

Several of the methods described here are applicable in the determination of fluorine in fluorinated organics. Depending upon the operator's preference, any one of the recommended methods can be used with good accuracy. Three of the most promising methods are: (1) combustion of the sample in moist oxygen, converting the resulting fluorine to silicone tetrafluoride and hydrolyzing the latter to hydrofluoric acid before titrating with sodium hydroxide;

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(2) fusion of the sample in a platinum crucible with sodium potassium carbonate, dissolving this residue, precipitating as lead chlorofluoride and determining the fluorine gravimetrically or decomposing the lead chlorofluoride precipitate and titrating the liberated chlorine; (3) fusion of the sample with metallic potassium and titrating the fluorine content with standard thorium nitrate solution.

Of the three suggested methods the procedure in which fluorine is titrated with standard thorium nitrate appears to be the most convenient, more suitable, and the most accurate.

WADC TR 55-108

August 1955

SUBJECT: THE EMISSION SPECTROGRAPHIC ANALYSES OF  
TITANIUM METALS AND ALLOYS

INVESTIGATORS: E. M. DuBois  
Arno Tuteur  
Lt J. L. Mahan, 1/lt USAF

CONTRACT: AF 33(616)-2315

CONTRACTOR: Spectrochemical Laboratories, Inc.

ABSTRACT: A Quantometric Point to Plane technique has been developed which is capable of giving accurate results, with reasonable speed, on solid samples of titanium alloys.

A vapor Injection Solution technique has been developed which will give excellent precision, accuracy and speed.

A statistical evaluation has been conducted on the following spectrographic techniques:

1. Fusion Coating
2. Electrode Dried Solution
3. Porous Cup
4. Rotating Disc
5. Lucite Cup
6. Vapor Injection
7. Point to Plane

Duplicate wet-chemical analyses have been performed on all samples submitted, for each of the elements studied, and these results have been used to determine the accuracies of the various quantometric procedures.

## BIOCHEMISTRY

WADC TR 54-421

August 1954

SUBJECT: ANTIFUNGAL ANTIBIOTICS

INVESTIGATOR: Frank L. Howard

CONTRACT: AF 33(616)-154

CONTRACTOR: University of Rhode Island

ABSTRACT: One hundred forty-three candidate antibiotics furnished by twenty-one agencies were tested for their fungicidal activity. The molds specified and used were Aspergillus niger, A. terreus, Myrothecium verrucaria, and Chaetomium globosum. The difficulty to inhibit spores of Alternaria solani, Helminthosporium carbonum and Curvularia lunata were used for germination tests. While most of the compounds were natural antibiotics, others were synthetic organic chemicals. Evaluation of antifungal action was obtained by four techniques: toxic agar in petri plates, spore germination inhibition on glass slides, impregnated cellulose pads on seeded agar, and retention of tensile strength impregnated thread after exposure to molds.

The five most antifungal antibiotics were found to be: comirin, benzyl mucochlorate, endomycin, netropsin sulfate, and rimocidin. The Squibb compound, 2-pyridinethiol 1-oxide and its twelve salts, compared favorably with standard copper 8-quinolinolate as a mildew inhibitor without necessarily staining the cloth. A furfural derivative exhibited evidence of high fungicidal action.

WADC TR 55-508

February 1956

SUBJECT: INVESTIGATION OF SELECTED CHEMICALLY ALTERED COTTON MATERIALS

INVESTIGATOR: Earlane L. Hamilton

ABSTRACT: Existing natural cotton materials require the addition of an add-on fungicidal treatment when resistance to fungi is required. Cotton which has been altered chemically under controlled conditions shows inherent resistance to fungi. A material with the properties of cotton but showing high fungus resistance is needed for use where an add-on chemical fungicidal treatment is not acceptable. For this reason, acetylated and cyanoethylated cotton have been evaluated. Some of the advantages of chemically altered cotton fabric compared to cotton treated with an add-on fungicidal treatment are as follows: permanence and stability of physical characteristics after alteration, increased fungus resistance, non-toxic to humans, and ability to remain unaffected by leaching action of water.

Preliminary tests showed that acetylated cotton provided different degrees of fungus resistance as the amount of alteration varied. Work was initiated to find the level of alteration by acetylation which gave adequate resistance to fungi, yet retained the desired physical properties. The cotton acetylated at 12.6  $\pm$  0.4% level in an 8 ounce or lower weight fabric was found to be the best degree of alteration of those evaluated since it provided satisfactory fungus resistance with the least impairment of the desired physical strength properties. The 10 and 12 ounce fabrics altered at a 12.6  $\pm$  0.4% level did not have adequate fungus resistance.

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In addition to the chemical alteration of cotton by acetylation, there is the reaction of acrylonitrile with cellulosic fibers to produce a partially cyanoethylated cotton material. The degree of chemical alteration by cyanoethylation is measured by the percent nitrogen content of the cellulosic fibers after modification. Preliminary testing of cyanoethylated cotton in a 3.6 ounce weight fabric with a 3.6% nitrogen content showed that satisfactory fungus resistance could be obtained by cyanoethylation.

More extensive evaluations are needed before comparison of the two types of chemical alteration under study can be made.

### CERAMICS

WADC TR 54-33 Part 2

February 1956

SUBJECT: PRELIMINARY MICROSCOPIC STUDIES OF CERMETS AT HIGH TEMPERATURES  
INVESTIGATOR: Thomas S. Shevlin, Herbert W. Newkirk, E. George Stevens,  
Harold M. Greenhouse  
CONTRACT: AF 33(038)-16911  
CONTRACTOR: The Ohio State University Research Foundation  
ABSTRACT: SECTION 1 - CHANGE IN THE MICROSTRUCTURE OF SOME TiC BASE CERMETS,  
WITH TIME, AT HIGH TEMPERATURES

The effect of heat treatment in a vacuum or inert-gas atmosphere at 2000°F on the microstructure of Kentanium samples K-151-A and K-152-B is discussed. Equipment and techniques employed in carrying out the research are discussed in detail. The effects of a solution-reprecipitation mechanism are discussed and a tentative theory is advanced relating the observed changes in microstructure to the steep slope of the stress-rupture curves for these cermets.

#### SECTION 2 - STUDY OF SOME TiC BASE CERMETS BY MEANS OF HIGH TEMPERATURE X-RAY DIFFRACTION TECHNIQUES

High temperature x-ray diffraction studies on phases stable at 2000°F in samples K-151-A and K-152-B and evidence for the solid solubility of TiC in Ni at this temperature are presented. In addition, the thermal expansion data of Ni from room temperature to 2000°F has been determined by high temperature x-ray techniques. The equipment used and the techniques employed in this research are discussed in detail. Evidence is presented which indicates that a solution-precipitation mechanism is responsible for the microstructure changes observed and reported in Section 1.

#### SECTION 3 - ELECTRON MICROSCOPY OF SOME TiC BASE CERMETS

A new electron microscopy replication technique is presented and its application in resolving the microstructure of sample K-152-B is discussed. The colloidal dispersion present in the Ni phase of this cermet have been resolved and found to be small particles of TiC. The origin of this precipitate and its effects on the high temperature strength of TiC-Ni cermets are discussed.



#### SECTION 4 - COEFFICIENT OF EXPANSION STUDIES ON HIGH TEMPERATURE ALLOYS

The relationship between thermal shock resistance and the linear thermal expansion of the phases composing a cermet is discussed. Techniques and apparatus for fabricating and testing alloys having thermal expansion characteristics similar to titanium carbide are presented. Thermal expansion data for the systems Fe-Co-Ni, Fe-Co-Ni-Cr, Cr-Mo-Ni, TiC+Fe-Co-Ni and TiC+Fe-Co-Ni-Cr are listed.

#### SECTION 5 - PREPARATION OF HIGH Ni-LOW TiC CERMETS FOR IMPACT AND THERMAL SHOCK STUDIES

Techniques and apparatus for the fabrication of TiC-Ni cermets suitable for impact and thermal shock studies and containing 73.1% Ni, 80.9% Ni, 87.9% Ni and 94.2% Ni by weight are given.

#### SECTION 6 - INVESTIGATION OF TiB<sub>2</sub> AND MoSi<sub>2</sub> AS THE CERAMIC COMPONENT OF A CERMET

The possibility of using a mixture of TiB<sub>2</sub> and MoSi<sub>2</sub> as components of a new high temperature cermet is discussed.

#### SECTION 7 - STUDY OF THE EFFECT OF HEAT TREATMENT ON IMPACT STRENGTH OF K-151-A AND K-151-B

The effect of heat treatment of the impact strength of Kentanium samples K-151-A and K-151-B is discussed. Apparatus and techniques employed in this research are discussed in detail.

WADC TR 54-467

July 1955

SUBJECT: REFRACTORY MATERIALS FOR USE IN HIGH TEMPERATURE AREAS OF AIRCRAFT  
INVESTIGATOR: Norman R. Thielke  
CONTRACT: AF 33(616)-199  
CONTRACTOR: The Pennsylvania State University  
ABSTRACT: The fracture patterns of TiC-Cermet stator blades which had been exposed to a simulated service test and the microstructures of these materials were investigated. An attempt to correlate fracture patterns and microstructures was unsuccessful.

The oxidation behavior of cermets was studied theoretically and experimentally. Theoretically, the tendency to oxidize was derived from an oxidation free energy plot for the reactions representing the complete oxidation of Ti-, Nb-, Ta-, Si-, Fe-, Co-, Ni- and Cr-carbides. Experimentally the oxidation behavior of carbide and metal powders was investigated using the differential thermal analysis technique. The oxidation resistance of carbide powders increased approximately in the following order: VC, TiC, NbC, B<sub>4</sub>C, WC, ZrC, TaC, Cr<sub>x</sub>C<sub>y</sub>, SiC. A similar classification has been found for metal powders with Cr and Si ranking best.

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The ternary system TiC-TaC-NbC has been investigated, using sintering temperatures up to 2530°C. The reactions were almost complete, indicating a complete solid solution throughout the system. Of all the sintered compositions in this system, TiC exhibited the greatest oxidation resistance.

The sintering and shrinking behavior of oxide mixtures simulating the oxides which are being formed during the oxidation of carbide Cermets, has been studied in search for compositions which form a dense and adherent oxide film.

Aluminum titanate bodies with additions of alumina, manganese carbonate and cesium Pyrex glass showed a modest increase in strength which was offset by an increase in thermal expansion.

An analysis of the applicability of thermal shock tests has been made. It follows that such tests are indispensable for the empirical determination of the maximum stresses as well as for a check on the theory of thermal shock. However, they are not suited as material tests.

A static method to measure the two thermal stress resistance factors  $R$  and  $R'$  is described and measurements on porous and dense titania, steatite, cordierite and  $\beta$  spodumene bodies are presented.

WADC TR 54-491

August 1956

SUBJECT: INVESTIGATION OF CARBON(GRAPHITE) BASE MATERIALS SUITABLE FOR ROCKET AND RAM-JET APPLICATIONS

INVESTIGATOR: William D. Smiley

CONTRACT: AF 33(616)-2023

CONTRACTOR: Stanford Research Institute

ABSTRACT: The details of the preparation, application, and testing of high temperature, oxidation and erosion resistant materials for nozzles are reported. A composition whose starting components were 4 mol percent molybdenum - 52 mol percent silicon - 44 mol percent carbon and which was prepared from a mixture of 75 weight percent silicon carbide and 25 weight percent molybdenum disilicide, has shown promising high temperature properties, including good oxidation resistance and refractoriness. Solid nozzles have been prepared from this material, together with liners for graphite nozzle, for testing of the WADC rocket test station.

WADC TR 54-491 Part 2

November 1955

SUBJECT: INVESTIGATION OF CARBON(GRAPHITE) BASE MATERIALS SUITABLE FOR ROCKET AND RAM-JET APPLICATIONS

INVESTIGATOR: William D. Smiley

CONTRACT: AF 33(616)-2023

CONTRACTOR: Stanford Research Institute

ABSTRACT: The details of the laboratory preparation and laboratory performance of carbon base materials developed for rocket nozzle applications are reported. Rocket nozzles were fabricated from these materials for evaluation at the WADC and Bell Aircraft Corporation rocket test stands.

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The most promising nozzle material developed in this program is zirconium-impregnated graphite. The formation of an adherent refractory skin of zirconium dioxide on the surface during operating service is responsible for the dimensional stability of this type of nozzle.

WADC TR 55-327

December 1955

**SUBJECT:** AN INVESTIGATION OF THE FEASIBILITY OF PRODUCING METAL BONDED CARBIDE BODIES BY THE EXTRUSION PROCESS  
**INVESTIGATOR:** William W. Wellborn  
**CONTRACT:** AF 33(616)-229  
**CONTRACTOR:** Firth Sterling, Inc.  
**ABSTRACT:** The main purpose of this investigation was to determine if useful metal-bonded carbide objects could be produced by the extrusion process. A further aim of this investigation was the comparison of the properties of such objects as might be produced by extrusion, with the properties of objects of the same composition produced by pressing, shaping, and sintering. The principal equipment used was a 1050-ton extrusion press, which had been built to extrude carbide charges.

The investigation revealed that it was possible to extrude these bodies into some shapes. However, shapes with irregular cross sections, or thin sharp edges, were extremely difficult to extrude and it was impossible to hold size. The physical properties of extruded materials were found to be measurably lower than materials compacted by conventional means. The incidence of flaws in extruded material was found to be greater.

#### COATINGS

WADC TR 54-325 Part 2

March 1956

**SUBJECT:** RESEARCH FOR LOW APPLICATION TEMPERATURE, ELECTRICALLY CONDUCTING, TRANSPARENT COATINGS FOR AIRCRAFT WINDSHIELDS AND RELATED COMPONENTS  
**INVESTIGATOR:** Paul Schall, Jr., Edwin H. Layer, Leonard H. Mauk, Robert E. Barrett, Edwin H. Lougher, Earl R. Olson, Harvey L. Goering  
**CONTRACT:** AF 33(616)-2467  
**CONTRACTOR:** Battelle Memorial Institute  
**ABSTRACT:** Transparent, conducting films of indium oxide on glass were prepared by thermal oxidation (at 250 to 400 F) of evaporated and sputtered indium films. Addition of tin to the films decreased their electrical resistance. Tin-doped films were prepared with resistance as low as 50 ohms per square and with optical transmittance of 75 to 80 per cent. The films are photoconductive. However, for the low resistance films (less than 100 ohms per square) the resistance changes only a few per cent when the films are light-dark cycled.

The temperature coefficient of resistance of the films is less than  $\pm 0.04$  per cent per degree (F).

Stability tests were conducted on the films under load (up to 1500 watts per square foot) and without load over a wide range of temperatures. Film resistance changes are less than 20 per cent in 150 hours.

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SUBJECT: DEVELOPMENT OF AN ORGANIC LACQUER RESISTANT TO FUMING NITRIC ACID  
INVESTIGATOR: Francis J. Honn, Robert E. Martin, David R. Wolf  
CONTRACT: AF 33(038)-21253  
CONTRACTOR: The M. W. Kellogg Company  
ABSTRACT: The objective of this contract was the development of a protective coating and sealant for the metal surfaces in the acid tank and ATO compartments of the B-47 aircraft. The evaluation of available coatings indicated polychlorotrifluoroethylene (KEL-F) was the only organic coating material with adequate resistance to fuming nitric acid. However its use was deemed impractical because of the difficulty of application on aluminum aircraft structures.

Research on the modification of KEL-F through copolymerization with vinylidene fluoride ultimately led to the development of Kellogg X-200 resin. Laboratory tests indicate that coating systems of X-200 filleting material and X-200 lacquer are adequate for the intended application. Coatings formed from the X-200 lacquer are far superior, in fuming nitric acid resistance, to any other lacquer coating evaluated.

SUBJECT: THE DEVELOPMENT OF A PROTECTIVE COATING RESISTANT TO NITRIC ACID AND HYDROCARBONS  
INVESTIGATOR: D. F. Siddall, H. L. Cahn, E. Hillier, M. Gunther  
CONTRACT: AF 33(616)-150  
CONTRACTOR: The United States Stoneware Company  
ABSTRACT: Numerous film-forming raw materials from solvent systems and nonvolatile filleting compounds have been screened for their resistance to white fuming nitric acid. Results show Fluorocarbon Resin X-200 from M. W. Kellogg Company is the only available resin meeting this contract requirement for coating and filleting compounds. Air dry, force dry or low temperature bake coatings have been tentatively formulated to provide the required protection. Studies of physical and chemical properties in conjunction with the compounding of this resin indicates the need for additional work to provide a more practical drying cycle and work is being continued concerning this phase.

Filleting compounds have been developed and tested to meet basic requirements. Further studies are being made with regard to equipment and manufacturing methods. Work on all phases, including the refinement of coating, production procedures on filleting compounds, and final cycling tests are to be carried out.

#### CORROSION

SUBJECT: PROPERTIES OF FUMING NITRIC ACID  
INVESTIGATOR: Webster B. Kay  
CONTRACT: AF 33(038)-10381  
CONTRACTOR: The Ohio State University Research Foundation

**ABSTRACT:** The equilibrium decomposition pressure of samples of  $\text{HNO}_3\text{-H}_2\text{O}$  mixtures containing 2.50 and 5.00 weight percent water and of  $\text{HNO}_3\text{-NO}_2$  mixture containing 7.50 and 15.00 weight percent nitrogen dioxide were determined between  $85^\circ$  and  $150^\circ\text{C}$  and at vapor volume to total volume ratios varying from 0.05 to 0.8. The decomposition measurements were carried out in a glass tube in a high pressure apparatus under isochoric conditions and with continuous stirring.

The experimental data have been expressed graphically a) as plots of equilibrium pressure vs ratio of vapor volume to total volume,  $V^G/V$ , at constant temperature and b) as plots of the equilibrium pressure vs the specific volume of the mixture at constant temperature.

By appropriate cross-plots of the data a series of diagrams showing the equilibrium pressure vs temperature at constant  $V^G/V$  ratios for integral values of initial water and nitrogen dioxide content were prepared and high temperature data extrapolated to  $25^\circ\text{C}$ .

The kinetics of decomposition of nitric acid were studied by analyzing available pressure-time rate data for different  $V^G/V$  values and in the temperature range from  $76^\circ$  to  $105^\circ\text{C}$ . Rate measurements were also carried out in the presence of excess nitrogen dioxide and water for different  $V^G/V$  values at  $85^\circ\text{C}$  and with excess oxygen at an initial pressure of 454 psig and at  $76^\circ\text{C}$ .

Under the conditions of the investigation, the decomposition of nitric acid is believed to be a homogeneous liquid-phase reaction.

The rate of decomposition can be represented satisfactorily by the equation.

$$\frac{dP}{dt} = Ae^{B(P^\infty - P)}$$

Where  $P^\infty$  = equilibrium pressure,  $t$  = time,  $e$  = base of natural logarithms, and  $A$  and  $B$  are constants. This equation holds over 80% of the reaction. The initial fast rate is followed by a slower apparent-first-order rate with respect to time.

The initial rates are (a) unaffected by addition of up to 15% nitrogen dioxide or by excess oxygen at an initial pressure of 454 psig at  $76^\circ\text{C}$ , and (b) lowered considerably by the addition of small amounts of water. The specific first-order rates are increased by excess nitrogen dioxide and oxygen but are lowered by added water.

The experimental energy of activation was found to be 30 kcal/mole, independent of the  $V^G/V$  ratio. The pre-exponential factor for the Arrhenius equation was  $10^{14} \text{ sec}^{-1}$ .

A tentative mechanism has been suggested, consistent with the available experimental data.

July 1955

SUBJECT: EVALUATION OF CORROSIVITY OF ROCKET BLAST RESIDUES AND  
CLEANING METHODS TO MINIMIZE CORROSION

INVESTIGATOR: Alfred H. Pagano, 1/Lt , USAF

ABSTRACT: Five different cleaning methods were tested in an attempt to remove the residues from six different rocket type blasts on fifteen different metal finish combinations. Results of the test indicate that the use of the standard cleaning method prescribed in T. O. 1-1-1, and a similar type cleaning method tried during this program were most effective in removing residues produced from various rocket residues. The above two methods followed by steam cleaning proved very effective as opposed to steam cleaning alone which was not effective. It was also found that certain rocket blasts were more corrosive to the materials tested than others, and that several of the finishes tested held up very well against rocket blast and may be suitable for application on aircraft to prevent corrosion caused by rocket blast.

WADC TR 54-568

July 1955

SUBJECT: RESEARCH ON TREATED MAGNESIUM SURFACES

INVESTIGATOR: Stanley E. Rohowetz

CONTRACT: AF 33(616)-2032

CONTRACTOR: Bjorksten Research Laboratories, Inc.

ABSTRACT: Research for the development of an accelerated performance test for treated magnesium alloys included the following corrosion resistance tests:

- (1) pH increase in 1.0N KCl.
- (2) Open circuit potential comparisons.
- (3) Short circuit current comparisons.
- (4) Hydrogen evolution rates in 1.0N KCl (gasometric method).

The following tests of adhesion of zinc chromate primer (MIL-P-6889A) to the treated surfaces were also included:

- (1) Adhesion in shear (wrought alloys specimens).
- (2) Adhesion in tension (cast alloys specimens).
- (3) Impact, ultrasonic vibratory, and pressure-sensitive tape tests (qualitative).

The gasometric method was the most promising test for evaluation of corrosion resistance. It was critical in its evaluation if unprimed treated magnesium alloys within five hours and of primed specimens in seven

The Dow #7 acid chromate treatment gave lower hydrogen evolution rates than the Dow #12 alkaline anodic treatment on all alloys tested.

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In the shear and tension adhesion tests, the Dow #7 gave higher adhesion to the primer than the Dow #12.

Corrosion rates for unprimed treated specimens obtained from several weathering tests did not correlate with the rates from accelerated laboratory tests. The corrosion rate of the Dow #7 was nonlinear and increased after a given time due to depletion of the protective chromium ions. Painted specimen rates, however, were in agreement with the accelerated test rates and showed the superiority of the Dow #7 over the Dow #12.

The corrosion resistance of primed-lacquered specimens far exceeded the difference in resistance afforded by the various treatments.

WADC TR 55-109

October 1955

SUBJECT: CORROSION STUDIES IN FUMING NITRIC ACID  
INVESTIGATOR: Edson H. Phelps, Fredrick S. Lee, 1/Lt USAF; Raymond B. Robinson  
ABSTRACT: Tests were conducted to determine the corrosion behavior of Armco 17-7PH stainless steel alloy in red fuming nitric acid containing various amounts of hydrofluoric acid as an inhibitor. It was found that hydrofluoric acid additions to red fuming nitric acid markedly reduced the corrosion rate of the alloy. Inhibition was obtained in both liquid and vapor phases at 120°F and 160°F, for periods up to 42 days duration. It was found that a hydrofluoric acid concentration of 0.25% by weight was marginal for good inhibition, and there was some indication that a hydrofluoric acid concentration of 0.75% was optimum. The presence of glass in the system diminishes the inhibiting effect of the hydrofluoric acid.

The corrosion potential of aluminum and stainless steel electrodes was observed in fuming nitric acids with various nitrogen dioxide and water contents, and with added fluoride. It was found that water additions shift the potential of both aluminum and stainless steel in the anodic direction. Nitrogen dioxide content up to 30% did not produce a significant change in the potential of either electrode. Fluoride additions caused a definite anodic shift in the stainless steel potential, and had a tendency to shift the aluminum potential in the cathodic direction. The observed effects were correlated in a qualitative manner with the previously reported corrosion rate effects of the variables tested.

#### DESIGN CRITERIA

WADC TR 53-24 Part 3

January 1956

SUBJECT: INTERMITTENT STRESSING AND HEATING TEST OF AIRCRAFT STRUCTURAL METALS  
INVESTIGATOR: G. J. Guarnieri  
CONTRACT: AF 33(616)-2226  
CONTRACTOR: Cornell Aeronautical Laboratory, Inc.

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**ABSTRACT:** Additional data have been gathered as part of a continuing program to evaluate the effect of intermittent load and temperature upon the high-temperature creep and rupture properties of aircraft structural alloys. The results obtained for the four alloys of 24S-T3 aluminum, RC-70 and RC-130-A titanium, and 4130 steel, when considered with respect to data previously reported for FS-1H magnesium, 321 stainless steel, Inconel X, and N-155, further emphasize that each alloy system behaves in its own characteristic manner when subjected to intermittent temperature and load. Data of the type being accumulated are of value to design engineers in the sense that some guidance is provided in the application of available static creep and rupture data to the construction of parts for intermittent service conditions.

Emphasis in the test program is now turning towards consideration of effects of combined cycling of load and temperature inasmuch as the creep and rupture behavior has been established for eight alloys under the simpler conditions of constant temperature-constant load, constant temperature-intermittent load, and constant load intermittent temperature.

WADC TR 54-175 Part 2

January 1956

**SUBJECT:** NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURE PT 2 "Analysis of Notched-Bar Rupture Life in Terms of Smooth-Bar Properties"

**INVESTIGATOR:** Howard R. Voorhees, James W. Freeman

**CONTRACT:** AF 18(600)-62

**CONTRACTOR:** University of Michigan

**ABSTRACT:** Rupture lives of smooth and notched bars have been compared in tests on three heat-resistant alloys with conventional heat treatments. Additional rupture tests were run for two of the alloys using notched and smooth specimens from material cold rolled between solution and aging treatments. The method of notch preparation was demonstrated to have a large effect on notched-bar life for some conditions. Variable results were attributed to residual stresses remaining from the machining operation.

Other experimental data obtained on smooth bars for some alloys and conditions included short-time tensile properties, creep curves, relaxation characteristics, and rupture life when the stress was changed from one level to another during the test.

A definite qualitative agreement was noted between notch strengthening or weakening and the rate of experimental stress relaxation under creep conditions. A quantitative stepwise analysis was developed to compare notch rupture behavior with properties of smooth bars. This analysis was based on the postulate that for a material to be notch strengthened it must be able to relax high initial stress concentrations quickly before a major portion of the total life has been expended in fibers initially at stresses above the nominal stress applied to the specimen. Further, the effective stress in such fibers must eventually drop below the nominal stress for notch strengthening to occur.



Calculation steps in the proposed analysis are summarized on page ix.

Completed calculations for one condition of notch strengthening and one of notch weakening showed satisfactory agreement with experimental findings and indicates that stress redistribution by creep-relaxation could by itself explain the wide differences in notch behavior observed for different materials and test conditions.

Further work is indicated to ascertain influence of other factors and to establish the range of validity of the proposed analytical method. Suggested extensions include tests on flat notched bars and use of other types of alloys.

WADC TR 55-18 Sup 1

February 1956

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT  
INVESTIGATOR: B. B. Muvdi, G. Sachs, E. P. Klier  
CONTRACT: AF 33(616)-2362  
CONTRACTOR: Syracuse University  
ABSTRACT: In Part II of this report data on the low-cycle rotating beam fatigue tests performed on hydrogen embrittled specimens of one heat of 4340 steel are presented and evaluated. The specimens were cathodically embrittled in a 10 percent sodium hydroxide solution, and tested on an R. R. Moore fatigue machine operated at approximately 250 rpm. All tests were limited to cycles ranging between 10 and 10,000.

The present report comprises a continuation of the work presented in Part I and as such it concerns itself with the same general purposes, namely, 1) the evaluation of hydrogen embrittlement of a steel heat treated to high strength levels and 2) the determination of suitable and sensitive means of evaluating such brittleness. While the low-cycle fatigue test reported here yielded valuable information, it is in no way as economical and sensitive as the static bend test reported in Part I.

WADC TR 55-103

January 1956

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS Pt 1 EFFECTS OF A NUMBER OF VARIABLES ON THE MECHANICAL PROPERTIES OF AIRCRAFT HIGH-STRENGTH STEELS  
INVESTIGATOR: G. Sachs, E. B. Muvdi, E. P. Klier  
CONTRACT: AF 33(616)-2362  
CONTRACTOR: Syracuse University  
ABSTRACT: This report presents a large amount of test data on a number of low-alloy steels, heat treated to strength values between 210,000 and 290,000 psi. The steels investigated were four heats of 4340 and one heat of each of the following: V-Mod. 4330, 98B40, Hy-Tuf, Super Hy-Tuf, Super TM-2 and Inco.

WADC TR 53-373 Sup 3

Furthermore, the results of tests previously performed on an additional heat of 4340 (small dia. bar) are included for purposes of comparison and completeness. The tests performed were the following: a) Tensions, which yielded information concerning the tensile and yield strengths as well as the ductility (reduction of area and elongation) of the steels studied. b) Notch-Tension, from which the notch strength, notch-strength ratio and information on the notch sensitivity of the steels were obtained. c) Impact, which permitted evaluation of impact characteristics of the steels at various test temperatures, as well as some information regarding the transition from impact-ductile to impact-brittle behavior. d) Fatigue and Notch-Fatigue, from which the endurance limit and the fatigue strength at various numbers of cycles were obtained for both smooth and notched specimens. e) Stress-Rupture, which permitted investigating the behavior of high-strength steels under sustained load conditions.

In addition, hardness measurements as well as metallographic studies were performed on all steels.

This report comprises first an extensive discussion of the effects of all fundamental factors investigated here. These factors are: (a) specimen position, (b) various heats of 4340 steel, (c) as-processed section size, (d) as-tested section size, (e) tempering temperature, (f) 500°F temper brittleness, (g) stress concentration, (h) direction-ality, (i) eccentricity, (j) loading time and (k) test temperature. This is followed by a comparison of the properties of the various steels, those of 4340 steel being used as basis.

The appendixes (Supplement 1 and 2) assembles the individual data in graphical and tabular form. Various parametric representations are used to facilitate their utilization.

WADC TR 55-103 Sup 1

January 1956

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS EFFECTS OF A NUMBER OF VARIABLE ON THE MECHANICAL PROPERTIES OF AIRCRAFT HIGH-STRENGTH STEELS

INVESTIGATOR: B. B. Muvdi, E. P. Klier, G. Sachs

CONTRACT: AF 33(616)-2362

CONTRACTOR: Syracuse University

ABSTRACT: Supplement 1 of WADC TR 55-103, is a summary of all test results assembled in this investigation. The results are presented in graphs which indicate all the important parameters.

SUBJECT: DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS Sup 2 Effects of a Number of Variables on the Mechanical Properties of Aircraft High-Strength Steels

INVESTIGATOR: B. B. Muvdi, E. P. Klier, G. Sachs

CONTRACT: AF 33(616)-2362

CONTRACTOR: Syracuse University

ABSTRACT: Supplement 2 of WADC TR 55-103 is a summary of all test results assembled in this investigation. The results are presented in tables which indicated both individual and average test values.

SUBJECT: MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS

CONTRACT: AF 33(616)-2303

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: The research program described in this report was undertaken to review the field of materials-property design criteria for metals used in aircraft and missiles, and to bring up to date a compilation of information of maximum or typical value for the design of aircraft and missiles. The information obtained in this study has been presented in such form as to be directly applicable to Bulletin ANC-5 (issued by the Air Force-Navy-Civil Panel on Strength of Metal Aircraft Elements.)

A great deal of work along this line has been done by the Air Force and other agencies. Much of this information has been published in WADC technical reports.

In the past few years, new metals and alloys have been developed, new properties were recognized as being important in design, and new information was obtained on the design uses of material properties. While much of this information is available in such sources as WADC technical reports, some is less readily available for the designer, and some gaps and inconsistencies in the existing information are apparent.

As a result of mutual agreement between representatives of the Materials Laboratory of Wright Air Development Center and Battelle Memorial Institute, investigation of seven specific subjects was undertaken. These are:

- (1) The development of recommended design values at elevated temperatures for SAE 4130, SAE 8630, 17-7(301), and 18-8 (302) steels.
- (2) A study of available data on bending moduli of steel and aluminum round tubes.

- (3) A study of available data on torsional modulus of rupture of steel and of aluminum round tubing.
- (4) A study of fatigue data on magnesium alloys in ANC-5.
- (5) A survey of available data on several stainless steels and superalloys.
- (6) A study of available data on spot-welded joints at depressed and elevated temperature.
- (7) Aluminum- and magnesium-base alloys for sand-, permanent-mold-, and die-casting applications.

Since the various phases were initiated and carried out separately, each will be reported in a separate section of this Final Report.

WADC TR 55-150 Part 2

November 1955

SUBJECT: MATERIALS PROPERTY DESIGN CRITERIA FOR METALS Pt 2 A STUDY OF METHODS OF PRESENTING CREEP DATA FOR AIRFRAME DESIGN

INVESTIGATOR: W. S. Hyler, H. J. Grover

CONTRACT: AF 33(616)-2303

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A study was made of a number of methods of presenting creep data from the standpoint of usefulness to the designer of airframes. These methods included: stress-time graphs, stress-temperature graphs, Larson-Miller graphs, and isochronous stress-strain graphs. Consideration was given to current practices and needs of personnel in the aircraft structures field. Also, consideration was given to the use of creep data in particular problems associated with high-speed flight.

On the basis of this study, it appears that a particularly useful method of presenting creep data is that of the stress-temperature graphs (with time as a parameter). It is also believed in some applications that the isochronous stress-strain graph may be a particularly desirable presentation, since useful engineering approaches for certain high-temperature problems have been advanced based on these graphs. This latter presentation should be considered tentative pending verification of these engineering approaches by experimental studies.

WADC TR 55-199 Part 1

August 1955

SUBJECT: TENSILE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING

INVESTIGATOR: Clifford L. Dotson, J. Robert Kattus

CONTRACT: AF 33(616)-424

CONTRACTOR: Southern Research Institute

WADC TR 53-373 Sup 3

**ABSTRACT:** The purpose of the work described in this report was to determine the effects of the following variables on the tensile properties of seven aircraft structural sheet metals after they had been heated within 10 seconds to temperatures up to 1200°F:

1. Strain rates from 0.00005 in. / in. / sec. to 1.0 in. / in. / sec.
2. Holding times at test temperature from 10 seconds to 30 minutes.

Special testing apparatus for heating and controlling the temperature of the test specimens, loading the test specimens, and recording load-strain curves was developed.

The test results showed that the tensile properties of the structurally stable test alloys - Type 321 stainless steel, RC-70A titanium and RC-130A titanium alloy - were not appreciably affected by changes in holding time. Because of the structural changes which occurred, the tensile properties of the precipitation hardening test alloys - Alclad aluminum alloys 2014-T6, 2024-T3, and 7075-T6 - and hard rolled AZ-31 magnesium alloy changed as the holding time was varied at certain temperatures.

With the exception of the results at one particular test condition, the strength properties of each alloy increased with increasing strain rates. The exception occurred in Alclad 2024-T3 alloy at 450°F, at which temperature a strongly mitigating structural change occurred. Percent elongation in the different test alloys varied inconsistently with increasing strain rates dependent upon the alloy, temperature, and holding time.

WADC TR 55-372

October 1955

**SUBJECT:** THE EFFECT OF SURFACE PREPARATION AND CONDITION ON MICROHARDNESS

**INVESTIGATOR:** F. H. Vitovec, H. F. Binder

**CONTRACT:** AF 33(616)-2803

**CONTRACTOR:** University of Minnesota

**ABSTRACT:** Observations on the load dependence of the pyramid microhardness are discussed and various explanations of it are briefly reviewed. Experimental results are presented which indicate the effect of the polishing procedure on the hardness-load relationship. A simplified mathematical analysis is presented which shows the relationship between the hardness-load behavior and the stress-strain curve under uniaxial deformation. The various hardness-load trends are explained in terms of strain hardening, effect of the free surface, and cold working introduced by the polishing procedure.

WADC TR 53-373 Sup 3

SUBJECT: AN INVESTIGATION OF THE INTERCHANGE OF TENSILE CREEP FOR COMPRESSIVE CREEP Pt. 1 Types 2024-T4 And 1100-0 Aluminum

INVESTIGATOR: O. K. Salmassy, R. L. Carlson, R. J. MacDonald, G. K. Manning

CONTRACT: AF 33(616)-2738

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Aluminum 1100-0 and aluminum alloy 2024-T4 were subjected to tension- and compression-creep testing at varying stress levels and temperatures. Creep data were compared to establish whether significant differences existed between tension and compression behavior. Room-temperature and elevated-temperature static properties of each material were obtained. Metallographic and hardness studies were used to supplement the results of creep and static tests. Test equipment and test techniques were developed which permitted creep measurements approaching 10 microinches per inch in sensitivity and  $\pm 25$  microinches accuracy. Data obtained on 2024-T4 and 1100-0 aluminum in all instances indicated greater creep resistance in compression. Differences in tension- and compression-creep strain decreased with increasing temperatures. Results indicated that a reversal in the interchange of 2024-T4 may take place between 375°F and that interchange may be a temperature-dependent phenomenon.

#### ELECTRODEPOSITION

WADC TR 53-162 Part 2

November 1954

SUBJECT: ELECTRODEPOSITION OF TITANIUM

INVESTIGATOR: Albert W. Schlechten, Martin E. Straumanis, C. Burroughs Gill

CONTRACT: AF 33(616)-75

CONTRACTOR: Missouri School of Mines and Metallurgy

ABSTRACT: A process has been developed for obtaining a diffusion coating of titanium on iron, mild steel, copper, and other metals. This coating is very high in titanium content and displays good corrosion resistance. Many of the variables in the process have been investigated and are reported. Data are also presented on the corrosion of titanium in fused salts, the resulting products, and the probable mechanism.

WADC TR 53-162 Part 3

March 1956

SUBJECT: ELECTRODEPOSITION OF TITANIUM

INVESTIGATOR: Albert W. Schlechten, Martin E. Straumanis, Sheng Tai Shih

CONTRACT: AF 33(616)-75

CONTRACTOR: Missouri School of Mines and Metallurgy

ABSTRACT: Processes are described by which diffusion coatings of titanium can be obtained on iron and steel objects. A fused chloride bath containing a titanium-oxygen alloy powder operated under helium was particularly effective. It was shown that the coating mechanism of this process was only partially due to the formation of titanium lower chlorides. Coatings were also obtained by deposition from the vapor state, using iodide or chloride vapors. The physical properties of the coatings are described.

## FATIGUE

WADC TR 54-531

May 1955

**SUBJECT:** INVESTIGATION OF MATERIALS FATIGUE PROBLEMS APPLICABLE TO  
PROPELLER DESIGN  
**INVESTIGATOR:** H. N. Cummings, F. B. Stulen, W. C. Schulte  
**CONTRACT:** AF 33(616)-493  
**CONTRACTOR:** Curtiss-Wright Corporation  
**ABSTRACT:** SAE 4340 steel, aircraft quality, from a single heat, at three high-hardness levels, was tested in rotating bending, and statistically determined S-N curves of constant probability of strength for smooth and for notched specimens were established. In addition, exploratory tests were made, by the Prot method, on the 4340 steel, and, by conventional methods, on ultra-high strength 4350 steel, for the mean endurance limit. The effect of inclusions on fatigue strength was studied, not only for the above tests but also for a small lot of vacuum-melted "inclusion-free" steel. A preliminary study was made of the possibility of using ultrasonic techniques for locating and measuring inclusions, and for detecting and measuring early fatigue cracks.

WADC TR 54-531 Sup 1

October 1956

**SUBJECT:** INVESTIGATION OF MATERIALS FATIGUE PROBLEMS APPLICABLE TO  
PROPELLER DESIGN  
**INVESTIGATOR:** H. N. Cummings, F. B. Stulen, W. C. Schulte  
**CONTRACT:** AF 33(616)-493  
**CONTRACTOR:** Curtiss-Wright Corporation  
**ABSTRACT:** WADC Technical Report 54-531 reported the fatigue characteristics of SAE 4340 steel as a guide for propeller and rotor designers. Stress levels for probabilities of 10, 50 and 90% survival under repeated loading were determined for a constant life. The knowledge, however, that 90% of a material will survive a given stress for, say, ten million cycles is not adequate for the design engineer. What he would like to know is at what stress 100 % of the material will survive. Therefore, the tests reported in WADC Technical Report 54-531 have been supplemented by additional fatigue tests in the long-life region in order to determine higher survival probabilities, in the order of 99%. Inclusion studies have revealed variability within the steel ingot that caused greater scatter in strength than was predicted by the tests reported in WADC TR 54-531.

WADC TR 55-86

January 1955

**SUBJECT:** FATIGUE CRACK DETECTION METHODS  
**INVESTIGATOR:** L. J. Demer  
**CONTRACT:** AF 33(038)-20840  
**CONTRACTOR:** University of Minnesota  
**ABSTRACT:** The factors involved in fatigue crack detection are discussed and the various nondestructive and destructive methods available for the purpose are described. Emphasis is placed on the use of these in laboratory fatigue tests on machined specimens. Numerous references are given both to literature dealing with the methods themselves and to investigations in which these methods have been employed. A critical evaluation is made of the various methods and the characteristic of each are set forth.

WADC TR 53-373 Sup 3

June 1955

SUBJECT: FATIGUE CRACK DETECTION METHODS

INVESTIGATOR: L. J. Demer

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: Current fatigue literature contains numerous references to a few recent studies in which fatigue cracks have been detected very early in the specimen life. This paper attempts to clarify such isolated observations by making a detailed review of the available experimental data in relation to crack initiation and growth in laboratory fatigue test specimens. The data are analyzed considering the nature of the test material, the character of the specimen, the stress level of the test, and the manner of testing. Conclusions are reached in regard to the effect of these factors on the percentage of fracture cycles at which fatigue cracks are detected. The formula of A. K. Head for the actual growth of fatigue cracks is reviewed and its agreement with experimental results is discussed.

WADC TR 55-225

June 1955

SUBJECT: STRENGTH, DAMPING, AND ELASTICITY OF MATERIALS UNDER INCREASING REVERSED STRESS WITH REFERENCE TO ACCELERATED FATIGUE TESTING

INVESTIGATOR: F. H. Vitovec, B. J. Lazan

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: The purpose of the work was to investigate the damping, stress-strain, and failure properties under uniformly increasing stress amplitude and to determine the relation of these properties to conventionally determined fatigue strength. Data are presented on SAE 1020 Steel, 24S-T4 Aluminum Alloy, SAE 4340 Steel, and RC-55 Titanium under rotating bending stress amplitudes which (a) are progressively increased during the test and (b) are held constant as in conventional fatigue tests. The Cough dynamic proportional limit method and the Lehr damping intercept under uniformly increasing stress amplitude were found to agree with the conventional fatigue strength only for certain materials and to be misleading in other cases. The failure stress at different rates of stress increase and different starting stresses was determined to evaluate the reliability of the Prot short-time fatigue testing method. For the materials tested Prot starting stress below the cyclic stress sensitivity limit (in the region where damping is unchanged by stress history) has practically no effect on the Prot failure stress. In general, the test results for the four materials indicate that the Prot method indicates the conventional fatigue strength with a reliability of 10 per cent. The use of modified Prot methods which utilize exponents  $n$  other than 0.5 were not significantly better for indicating fatigue strength.

WADC TR 53-373 Sup 3



**SUBJECT:** EFFECT OF CYCLIC LOAD FREQUENCY ON THE CREEP-RUPTURE AND FATIGUE PROPERTIES OF JET ENGINE MATERIALS

**INVESTIGATOR:** L. A. Yerkovich, G. J. Guarnieri

**CONTRACT:** AF 33(616)-42

**CONTRACTOR:** Cornell Aeronautical Laboratory, Inc.

**ABSTRACT:** An investigation has been conducted to evaluate the effects of cyclic loading and load frequency on the elevated temperature creep-rupture properties of several jet engine sheet materials. Specifically the behaviors of low carbon N-155, type 321 stainless steel and Inconel X were studied, when exposed to various combined steady and cyclic stresses at various stress amplitudes and temperatures within a wide range of test frequencies.

Data for selected static and dynamic test conditions are presented in various tabular and chart forms to illustrate the influence of direct fluctuating stresses on the creep and rupture characteristics of the test alloys. These data demonstrate that the static load high temperature creep and rupture behavior of N-155, type 321 stainless steel and Inconel X are not always altered by the superposition of cyclic stresses; however, damage may be accelerated or retarded depending upon temperature, static stress level and the frequency of the cyclic stress component.

WADC TR 55-456

October 1955

**SUBJECT:** RELATION OF INCLUSIONS TO THE FATIGUE PROPERTIES OF SAE 4340 STEEL

**INVESTIGATOR:** H. N. Cummings, F. B. Stulen, W. C. Schulte

**CONTRACT:** AF 33(616)-493

**CONTRACTOR:** Curtiss-Wright Corporation

**ABSTRACT:** From studies of an unusually large number of fatigue data, certain trends in the relation of inclusions to fatigue strength of SAE 4340 steel were brought to light. After discussing these trends in some detail, an explanation concerning the mechanism of fatigue is proposed and various predictions are made based on the proposed explanation.

WADC TN 55-232

June 1955

**SUBJECT:** ANALYSIS OF SLIP DAMPING WITH REFERENCE TO TURBINE BLADE VIBRATION

**INVESTIGATOR:** L. E. Goodman, J. H. Klumpp

**CONTRACT:** AF 33(038)-20840

**CONTRACTOR:** University of Minnesota

**ABSTRACT:** Energy of vibration may be dissipated by microscopic slip on interfaces where machine elements are joined in a press fit. In this report slip damping is studied as an agent in reducing turbine blade resonant stresses and prolonging turbine life. A general theory of slip damping is developed and an expression for the energy loss per cycle of oscillation is found. The predictions of the theory are compared with the results of controlled experiments. It appears that the theory is in satisfactory agreement with experiment and with measurements made on turbine blades elsewhere in this country and abroad.

The implications of the general theory in the design of turbine blades are discussed. It appears that slip damping is capable of being an effective agent in reducing resonant stresses, especially in the 'stall-flutter' condition where aerodynamic damping is inadequate. The design of a slip damping joint which would achieve theoretically possible energy decrements much larger than are present in existing commercial construction is shown to depend on the maintenance of an optimum contract pressure.

WADC TN 55-273 Part 1

June 1955

SUBJECT: CUMULATIVE FATIGUE DAMAGE OF AIRCRAFT STRUCTURAL MATERIALS  
Part 1: 2024 and 7075 Aluminum Alloy

INVESTIGATOR: A. M. Freudenthal, R. A. Heller, P. J. O'Leary

CONTRACT: AF 33(616)-2274

CONTRACTOR: Columbia University

ABSTRACT: The object of this investigation is to determine the effect of randomly varying stress-amplitudes representing gust or maneuver load sequences encountered in flight on the fatigue life of AA7075 and AA2024 aluminum by testing small rotating beam specimens subject to random time series of stress-amplitudes derived from specific frequency distributions of stress-amplitudes. The tests were carried out on specially built random fatigue testing machines of a type recently developed in the Civil Engineering Research Laboratories of Columbia University.

Over a thousand specimens were tested. Three sets of six stress levels were used for each material with which nine different load distributions were constructed. In addition, conventional constant amplitude tests were carried out to determine the S-N diagrams. The results were statistically analyzed and presented on the basis of two and three parameter extreme value distributions. While a detailed evaluation of the test results will be presented in the second part of this report, the tentative statement can be made that according to the test results at present available, only for the least severe stress distributions within the low range of stresses does the sum of the cycle ratios itself provide a rough estimate of the cumulative damage under varying stress-amplitudes.

WADC TN 284

September 1955

SUBJECT: EFFECT OF MATERIAL DAMPING AND STRESS DISTRIBUTION ON THE  
RESONANT FATIGUE STRENGTH OF PARTS

INVESTIGATOR: Egons R. Podnieks, Benjamin J. Lazan

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: The behavior of various types of parts under resonant vibrations is reviewed considering material hysteresis as the only form of damping present. Three different criteria are introduced for comparing materials and parts for

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resonant operation: total damping -  $D_d$  oc, resonance amplification factor -  $A_r$  ( $A_r'$ ), and resonance exciting stress -  $S_g$  ( $S_g'$ ). Graphical and analytical procedures are derived for determining the unitless damping energy oc and the unitless elastic strain energy  $E$ . Both relate the effect of the shape of the part and the stress distribution to the resonant behavior of a part. The relationship of stress distribution to the shape of the damping energy curves and the resultant effect of the above criteria is discussed. The influence of the cyclic stress history on resonant behavior is also indicated. In comparative examples the relative merits of five different types of structural materials (type 403 alloy, titanium - RC55 annealed, RC55 cold-worked, RC130B, and glass fabric laminate) based on above criteria are discussed in connection and with application to various types of parts. Resonant fatigue curves are introduced and discussed for the same examples.

WADC TN 55-460

September 1955

SUBJECT: EFFECT OF PRIOR CREEP ON THE REVERSED STRESS FATIGUE PROPERTIES OF TEMPERATURE RESISTANT ALLOYS

INVESTIGATOR: Franz H. Vitovec, Benjamin J. Lazan

CONTRACT: AF 33(616)-2803

CONTRACTOR: University of Minnesota

ABSTRACT: The purpose of this work was to investigate the effect of prior creep on the fatigue properties of the temperature resistant alloys, N-155, S-816, and Stellite 21. In order to separate the effects of stress, time and temperature variables, four different types of tests have been performed: conventional axial reversed stress fatigue tests, creep tests, Prot tests with specimens aged unstressed for various times, and Prot tests with specimens which were subjected to various amounts of creep prestrain.

Increasing amounts of prior creep caused first a slight increase and then a rapid decrease of the Prot fatigue strength of N-155 and S-816. For Stellite 21, the Prot failure stress decreased continuously with increasing prestrain due to aging effects during the creep prestrain. The criterion found to most accurately predict fatigue damage was the percent creep rather than the state of creep.

WADC TN 55-527

September 1955

SUBJECT: A REVIEW OF OBSERVATIONS ON THE CRACKING CHARACTERISTICS AND FRACTURES OF LABORATORY FATIGUE SPECIMENS

INVESTIGATOR: L. J. Demer

CONTRACT: AF 33(038)-20840

CONTRACTOR: University of Minnesota

ABSTRACT: This review examines experimental observations relating to characteristics of the fatigue cracking and fracture of laboratory specimens both of single crystals of metals and polycrystalline metallic structures of various crystal habits. The sections deal with the formation and the paths of fatigue macrocracks, the occurrence of fatigue macrocracks, the characteristics of fatigue fractures and certain associated phenomena, and the path of fatigue macrocracks under combined stresses. The object of the report is to provide background for further study of the mechanism of the fatigue process in metals.

FUR

JOINING

WADC TR 54-17 Part 2

November 1955

SUBJECT: JOINING OF MOLYBDENUM  
INVESTIGATOR: W. N. Platte  
CONTRACT: AF 18(600)-114  
CONTRACTOR: Westinghouse Research Laboratories  
ABSTRACT: A study of the factors which influence the physical properties, especially the ductility, of molybdenum welds is discussed in this report. Welds in both arc-cast and vacuum sintered molybdenum made in commercially pure inert atmospheres and in contaminated inert atmospheres are examined.

The effects of several deoxidizers on the welding properties of vacuum sintered molybdenum are examined. The specific requirements of deoxidizing agents are established and it is shown that titanium between 0.2 and 0.5% meets these requirements.

The effects of interstitial elements, oxygen, nitrogen and carbon, on the weld properties of arc-cast molybdenum have been examined. It is shown that the limits for oxygen in the inert gas welding atmosphere can be predicted. The deleterious effects of nitrogen and oxygen are shown quantitatively by means of bend test data. Both oxygen and nitrogen reduce the ductility of molybdenum weld metal. Carbon is also believed to have a similar effect.

Oxygen and nitrogen are shown to have an interaction effect when used in combination in the inert welding atmosphere. Nitrogen reduces the deleterious effects of oxygen and vice versa, but the combination is not as effective as high purity inert gas as a welding atmosphere.

Exploratory studies are described for slightly improved inert gas atmospheres and post-weld heat treatment. But welds were made to establish the validity of using "bead on plate welds" as a test criterion.

WADC TR 55-213

July 1955

SUBJECT: DEVELOPMENT OF BRAZING ALLOYS FOR JOINING HEAT RESISTANT ALLOYS  
INVESTIGATOR: Forbes M. Miller, Robert L. Peaslee,  
Homer S. Gonser  
CONTRACT: AF 33(616)-2287  
CONTRACTOR: Wall Colmonoy Corporation Research Laboratory  
ABSTRACT: Fifteen different alloy systems were tested and studied for brazing characteristics, chemical and physical properties. These alloys were nickel base binary and ternary systems containing such metals and metalloids as phosphorus, silicon, chromium, manganese, molybdenum, tungsten, and iron.

The nickel-phosphorus-chromium system and nickel-phosphorus-silicon system showed the best properties of the phosphorus bearing alloys. The nickel-silicon-manganese system showed the best properties of the silicon

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bearing alloys. Of the alloys studied the silicon bearing alloys in general are higher melting than the phosphorus bearing alloys.

Studies also showed that phosphorus and manganese contributed the most toward improving the flow and wetting properties of the alloys, while silicon and chromium contributed the most toward improving the oxidation resistance and strength properties of the alloys.

#### LEATHER

#### METALS

#### GENERAL

AFTR 6731 Part 4

January 1956

SUBJECT: SHORT TIME CREEP PROPERTIES OF STRUCTURAL SHEET MATERIALS FOR AIRCRAFT AND MISSILES

INVESTIGATOR: John A. Van Echo, Ward F. Simmons,  
Damian V. Gullotti, James R. Bibler,

CONTRACT: AF 33(038)-8743

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Creep properties are reported for several commercial sheet materials including RC-70 titanium, 61S-T6 and 75S-T6 aluminum alloys, SAE 4130 steel, the heat-resistant Alloy Steel A-286, and two stainless steels--17-7PH and Type 410. The test results were obtained under special short-time (1/2 to 120 minutes), rapid-heating (50 to 250 F per second) test conditions to simulate service conditions. Each alloy was tested over the useful temperature range for the particular material.

Applications of these alloys under special short-time conditions might include skin material for supersonic vehicles, combustion chambers for rockets and missiles, and other structural parts in aircraft and missiles.

This report is Part 4 of a project that was undertaken to obtain the creep properties of numerous sheet materials. Previous work was similarly done on a wide range of other commercial and some experimental sheet materials.

#### FERROUS

WADC TN 55-87

November 1955

SUBJECT: SOME EFFECTS OF SILICON ON THE MECHANICAL PROPERTIES OF HIGH STRENGTH STEELS

INVESTIGATOR: Chester H. Shih, Morris Cohen  
B. L. Averbach

CONTRACT: AF 33(616)-2012

CONTRACTOR: Massachusetts Institute of Technology

ABSTRACT: The effects of increasing the silicon content in 4340 and 4325 steels to 1.5 percent have been observed as a function of tempering temperature. The softening on tempering is retarded by silicon, and it appears possible to obtain somewhat higher strengths in the silicon steels without a corresponding loss in ductility or in Charpy impact strength. At high strength levels the elastic limit is approximately one-half of the engineering yield strength;

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silicon additions have no influence on the maximum elastic limit. Although the introduction of silicon has several beneficial effects at high strength levels, undesirable impact properties may be introduced by tempering at high temperatures (temper embrittlement). Retained austenite data are presented as a function of cooling rate from the hardening temperature, and it is shown that substantial amounts of retained austenite may be developed on retarded cooling. There is also some indication that the fatigue endurance limits and the elastic limits may be related.

#### HIGH TEMPERATURE

WADC TR 54-391 Sup 1

January 1956

SUBJECT: INVESTIGATION ON NOTCH SENSITIVITY OF HEAT RESISTANT ALLOYS AT ELEVATED TEMPERATURES-Sup 1 APPARENT VOLUME INCREASE DUE TO TRIAXIAL STRESSES

INVESTIGATOR: W. E. Dirkes, Capt, USAF

ABSTRACT: The volume of structural metals is generally considered to remain constant even with extremely high stresses applied. Some data recently published indicate significant changes in volume under stress-rupture test conditions for specimens having stress gradients. More recent analysis of the same data and specimens together with additional measurements from these specimens show that significant volume changes did not actually occur, but that plane cross-sections of specimens did not always remain plane when subjected to stress gradients.

WADC TR 55-218

September 1955

SUBJECT: INVESTIGATION OF NICKEL BASE PRECIPITATION HARDENING ALLOYS

INVESTIGATOR: David I. Sinizer

CONTRACT: AF 33(616)-2144

CONTRACTOR: National Research Corporation

ABSTRACT: Vacuum-melted Waspaloy was made with controlled carbon content and varying ratios of titanium to aluminum. All nominal compositions were within the chemical specification ranges for commercial Waspaloy. Standard melting and casting procedures were satisfactory, except that an unusually large hot top appears necessary for producing sound ingots. All ingots forged readily. Increasing Ti/Al ratio from about 1.5 to 3.0 had no appreciable effect on rupture life or minimum creep rate at 1500°F for stresses from 30,000 to 35,000 psi. Both elongation and reduction of area at failure appeared to decrease with increasing Ti/Al ratio. None of the alloys were notch sensitive in stress-rupture at 52,500 psi and 1350°F. There was an indication of a minimum notch sensitivity at a Ti/Al ratio of about 2.0 for both stabilized and unstabilized vacuum-melted Waspaloy.

WADC TR 55-388

January 1956

SUBJECT: A SURVEY OF THE EFFECT OF AUSTENITIZING TEMPERATURE AND RATE OF CONDITIONOUS COOLING ON THE STRUCTURE AND 700°F to 1200°F PROPERTIES OF THREE LOW-ALLOYED STEELS

INVESTIGATOR: Kenneth P. MacKay,  
A. Phillip Coldren,

Adron I. Rush,  
James W. Freeman

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CONTRACT: AF 33(038)-13496  
CONTRACTOR: University of Michigan  
ABSTRACT: The relationships between microstructures formed with various cooling rates and austenitizing temperatures and properties at 700° to 1200°F were surveyed for three low-alloyed steels. The steels were Ni-Cr-Mo-(SAE 4340), 1.25Cr-Mo-V ("17-22-A"S), and 3Cr-Mo-W-V(H-40). Martensite, martensitic-bainitic, bainitic, and bainitic-ferritic structures were produced by oil quenching 1-inch rounds and air cooling 1-inch and simulated 3-and 6-inch rounds. Modifications of the oil-quenched and normalized structures of the 1-inch rounds resulted when the austenitizing temperature was raised from 1750° to 2100°F. The hardness level was maintained at 280-320 BHN by tempering the structures which had higher hardnesses in the as-transformed condition.

The results indicated that the fully bainitic structures which were predominantly upper bainite had maximum strength over the range of testing temperature used. In general, such structures were found in the larger, normalized sections and with the higher austenitizing temperature. Since low values of ductility resulted from the higher temperature treatments, however, the best combinations of strength and ductility were obtained when the largest sections were normalized from the lower austenitizing temperatures (1750°F for SAE 4340 and "17-22-A"S, and 1950°F for H-40).

Regarding the effect of varying the cooling rates of normalized bars, it was found that increases in strength occurred for all three steels as the effective bar diameter was increased from 1 inch to 6 inches. The effect of raising the austenitizing temperature from 1750° to 2100°F was to increase the strength, with the H-40 steel being affected the most. Ductility was lowered for all three steels as the heat-treating temperature was raised.

WADC TR 55-470 Part 1

June 1955

SUBJECT: AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS

INVESTIGATOR: Clarence A. Siebert, Lynn H. De Smyter,  
Maurice J. Sinnott, Robert E. Keith

CONTRACT: AF 33(616)-353

CONTRACTOR: University of Michigan

ABSTRACT: Chromel alloys ASM, ARM, and D, and type 310 stainless steels were oxidized for 100-hour periods in the stressed condition. The above alloys and Inconel were oxidized for times up to 500 hours in the unstressed condition. Intergranular oxidation measurements were obtained microscopically. The influence of stress was to cause an increase in the intergranular penetration increased with increasing time and temperature. Increasing the water-vapor content of the air increased the intergranular penetration slightly. The effect of a preferred orientation decreased the intergranular penetration slightly. The weight gained during oxidation was determined. It was found that in the alloys tested that a plot of the square of the specific weight gain versus temperature resulted in a straight-line relationship. Visual and magnetic examinations were made on the oxidized specimens and their oxides. No correlation between these observations and oxidation properties could be determined. X-ray diffraction patterns were made on representative oxides. This analysis showed the scales encountered to be of a

protective nature by Randall and Robbs criteria of the presence of  $\text{Cr}_2\text{O}_3$  or high-parameter spinel. Electron diffraction examination of the subsurface structure was performed on type 310 stainless steels oxidized in the unstressed condition. It was found that the oxidation products in the subsurface region were substantially the same as the surface oxides as determined by x-ray diffraction techniques.

WADC TN 55-290

October 1955

**SUBJECT:** DEVELOPMENT OF CAST IRON-BASE ALLOYS OF AUSTENITIC TYPE FOR HIGH HEAT-RESISTANCE AND SCALE-RESISTANCE

**INVESTIGATOR:** F. Eberle, W. Feduska,  
W. E. Leyda, F. B. Snyder

**CONTRACT:** AF 33(616)-2413

**CONTRACTOR:** The Babcock & Wilcox Company Research Center

**ABSTRACT:** For the purpose of developing iron-base substitute alloys for the cobalt-base alloy H.S. 21. distinguished by

- 1) a minimum iron content of 45%
- 2) a fully austenitic microstructure,
- 3) adequate oxidation resistance at 2000°F,
- 4) a creep-rupture strength at 1600° and 1800°F similar to that of H.S. 21, and
- 5) suitability for fabrication by the investment-casting or shell-molding process,

over 100 investment-cast experimental compositions were studied. A base composition containing 45% Fe, 20% Cr, and 27% Ni was found to possess the required lattice structure and oxidation resistance and to retain these characteristics when strengthened by the addition of 4-7.5% of carbide- and compound-forming elements. For strengthening purposes, dual and triple additions of Ta, Cb, Ti, Zr, Be, Mo, and W, at concentration ratios of 1:1:1 (1:1), 2:1:1 (2:1), 1:2:1 (1:2), and 1:1:2 were investigated. The most-promising composition found was an alloy containing:

0.52C, 1.11Mn, 0.7Si, 19.8Cr, 27.8Ni, 2.02Cb, 0.63Ta, 2.2W, 0.05CoLa. Its rupture strength at 1600° and 1800°F was equal to about 80-85% that of H.S. 21. Preliminary tests indicated the possibility of raising the strength level of this alloy further by increasing the tungsten content relative to that of CbTa and by replacing part of the nickel by cobalt.

#### NONFERROUS - ALUMINUM

WADC TR 54-119

November 1955

**SUBJECT:** PROPERTIES OF XA78S ALUMINUM ALLOY SHEET, PLATE, AND EXTRUSIONS

**INVESTIGATOR:** H. W. Zoeller  
R. E. Wittman

**ABSTRACT:** The mechanical and metallurgical properties of XA78S aluminum alloy clad sheet, plate and extrusions have been evaluated in this work. Long time tests will be reported in a supplement to this work. The results when compared with the evaluation by other investigators, may lead to the acceptance of this alloy as an aircraft material. The XA78S aluminum alloy has approximately 10 percent greater strength than other high strength aluminum alloys. It has corrosion resistance, fatigue strength, microstructural characteristics, response to



heat treatment and spot welding properties comparable to other commercial aluminum base alloys containing zinc, magnesium, and copper. This alloy offers greater resistance to bending therefore, the forming will be more difficult. As a result of this evaluation three specifications have been issued. The designations for XA78S, 75S and 24S aluminum alloys have been recently changed to X7178, 7075, and 2024, respectively.

WADC TR 54-590

September 1955

SUBJECT: POWDER FABRICATION OF ALUMINUM ALLOYS  
INVESTIGATOR: J. B. Hess, R. S. Mateer  
CONTRACT: AF 33(616)-2296  
CONTRACTOR: Kaiser Aluminum and Chemical Corp.  
ABSTRACT: A fabrication method, combining powder metallurgy techniques and conventional extrusion, was developed for the purpose of alloying aluminum with refractory compounds and other unusual constituents. Additions of  $B_4C$ ,  $Al_2O_3$ ,  $TiC$ ,  $SiC$ ,  $ZrO_2$ ,  $WC$ ,  $TiAl$ ,  $MnAl_6$ ,  $FeAl_3$ ,  $Mo$ ,  $Cr$ ,  $Si$  and  $Cu$  were made to a base of commercial atomized aluminum powders, and the resulting alloy properties were determined.

Outstanding improvements in Youngs' modulus were achieved by means of the  $SiC$  and  $B_4C$  additions; however, accompanying tensile properties were only mediocre.

Current attempts to improve these tensile properties by utilizing prealloyed powders as bases for the refractory additions are still in preliminary stages of study.

WADC TN 55-49

September 1955

SUBJECT: THE EFFECT OF PRIOR CREEP ON THE MECHANICAL PROPERTIES OF ALCLAD 2024-T3  
INVESTIGATOR: Clark E. Beck  
ABSTRACT: Tensile tests at room temperature and at 500°F. were conducted on Alclad 2024-T3 (formerly designated as clad 24S-T3) aluminum alloy sheet specimens after they had been subjected to various amounts of creep deformation. This prior creep deformation was obtained by stressing the specimens at constant loads in tension for periods of approximately 20 hours and 90 hours. The amounts of prior deformation varied from 0% in 2 inches for no applied stress to 2% in 2 inches with a stress of 15,500 psi applied.

At room temperature the tests showed a decrease in ultimate tensile strength properties as the amount of prior creep deformation increased toward the maximum values considered in this investigation. However, at 500°F the tensile yield strengths were much nearer the ultimate tensile strengths on a percentage basis than was shown for the room temperature tensile tests. There was a notable decrease in ultimate tensile and tensile yield strength values as

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the amount of prior creep deformation increased. This was particularly true for those specimens in which the prior deformation had been obtained in 90 hours.

NONFERROUS - MAGNESIUM

October 1955

WADC TR 55-160 Part 1

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES  
Pt: 1 Improved Mg Wrought Alloys  
INVESTIGATOR: J. D. Wood, 1/Lt., USAF  
CONTRACT: AF 33(616)-2337  
CONTRACTOR: The Dow Chemical Company  
ABSTRACT: The work on this contract was directed toward the improvement of high strength wrought Mg alloys through alloy survey based on Mg-Zn and evaluation of promising compositions in pellet fabrications. A Mg sheet alloy containing 4-5% Zn, 1-2% MM, and 1-2% Mn with an excellent combination of workability, strength, and ductility was developed. A typical yield strength of 35,000 psi or higher should be realized through proper fabrication of these alloys. Further study of the Mg-Ag-Zn-Zr system indicated that high strength sheet can also be obtained by work hardening Mg-Ag-Zn-Zr alloys such as QZ66 and QZ63. Additional work is necessary to determine if higher strength can be obtained in these alloys than in the Mg-Zn-MM-Mn (or Zr) system. Considerable promise is also shown by HM31 pellet extrusions. Pellet fabrication of this alloy substantially increases static strength, particularly CYS at temperatures up to 600°F. It has the highest static strength in the range of 300-600 F of any Mg extrusions yet developed.

WADC TR 55-160 Part 2

October 1955

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES  
Pt. 2 Physical Properties of Mg Base Alloys  
INVESTIGATOR: J. D. Wood, 1/Lt., USAF  
CONTRACT: AF 33(616)-2337  
CONTRACTOR: The Dow Chemical Company  
ABSTRACT: The coefficients of linear thermal expansion have been determined for pure magnesium, AZ31A, EZ33A, HK31XA, and HZ32XA using a high precision dilatometer. The electrical conductivities of AZ31A, AZ31B, AZ63A, AZ92A, cast ZK60A, and pellet extruded ZK60A have been determined using a Kelvin Bridge. Values obtained compare favorably with previous determinations. Thermal conductivities of the alloys were calculated from the electrical conductivity data using the Powell and Bungardt equations. The heat capacity and heat fusion of pure magnesium are also reported.

The effects of heat treatment, composition, mechanical history, and working direction on electrical conductivity were noted. There was no significant effect of these variables on thermal expansion.

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November 1955

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES Pt. 3  
INVESTIGATOR: J. D. Wood  
CONTRACT: AF 33(616)-2337  
CONTRACTOR: The Dow Chemical Company  
ABSTRACT: Inhomogeneous deformation has been studied in high-purity magnesium and magnesium-calcium alloys. Metallographic examination of cold-rolled sheet disclosed that narrow bands of preferential recrystallization had occurred in the alloys containing less than 0.055 percent calcium. These bands rotated toward the rolling plane with increasing reduction while newly formed bands subtended an angle of 30°. Room temperature compression tests on hot-rolled materials showed that the magnesium showed banding whereas a Mg-0.5 Ca alloy did not, in agreement with the observations on sheet. The homogeneity of deformation also showed an isotropy in the compression tests. In both compression and rolling the bands became eventual crack loci.

A large amount of information on preferred orientation of flat magnesium alloy extrusions has been obtained. As in the case of rolled magnesium alloy sheet, various deviations from the ideal orientation occur. These deviations are primarily rotations of the maximum basal pole density about the transverse direction as an axis. In addition, marked variation of preferred orientation with depth below the surface has been found. Profile plots of the rotation as a function of depth fall into two broad groups: (1) those which show a double orientation at the center and (2) those which show a single orientation at the center. A theory is outlined in an attempt to explain these general categories of sheet and flat extrusions. Basal slip and twinning are assumed of primary importance. Compressive strains are modified by shear strain in such a way as to explain many of the experimental observations in a consistent manner.

#### NONFERROUS - TITANIUM

WADC TR 54-109 Part 2

November 1955

SUBJECT: SCALING OF TITANIUM AND TITANIUM ALLOYS  
INVESTIGATOR: H. J. Siegel, R. C. Duncan, Jr., R. E. Swift  
CONTRACT: AF 18(600)-60  
CONTRACTOR: University of Kentucky  
ABSTRACT: A preliminary study of the scaling characteristics in air of experimentally produced titanium and titanium-base alloys was conducted at temperatures of 1200°, 1400°, 1600°, and 1800°F (650°, 760°, 870°, 980°C) in the time range of approximately four to three hundred hours. A total of thirty-six titanium-base alloys were scaled at each of these temperatures. Scales formed on all of the alloys at 1600°F were studied using x-ray diffraction. Scaling propensity of titanium-base alloys relative to unalloyed titanium were evaluated on the basis of weight gain with time. Isothermal transitions were noted for many of the alloys but were not studied in detail. An investigation of gaseous penetration in the commercial materials RC-70, RC-130A, and RC-130B was made at 1600° and 1800°F.

June 1955

SUBJECT: TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATIONS  
INVESTIGATOR: W. F. Carew, D. J. McPherson, F. A. Crossley  
CONTRACT: AF 33(038)-22806  
CONTRACTOR: Armour Research Foundation of Illinois Institute of Technology  
ABSTRACT: Results on the following phases of the investigation are presented: (1) effect of carbon, nitrogen and oxygen on the tensile, impact and creep-rupture properties of Ti-6% Al alloy; (2) tensile test evaluation of experimental alloys including binary alloys of stabilizing additions to titanium; (3) effect of vacuum annealing on tensile ductility of alloys containing 8% or more of aluminum; (4) creep-rupture evaluation of alloys; (5) creep evaluation of alloys; (6) evaluation of stability of room temperature tensile properties upon exposure to creep at elevated temperatures; and (7) evaluation of the rolling and welding characteristics of the alloys: 6% Al-0.5% Si, 6% Al-4% V and 7% Al-3% Mo.

The additions carbon, nitrogen and oxygen were found to improve tensile strength of the 6% Al alloy at temperatures to 500°C, the highest test temperature. Additions of 0.3% nitrogen and 0.5% oxygen produce complete embrittlement at room temperature. All impact values were low and it is believed that this was due to hydrogen contamination. The additions improved creep and rupture performance of the 6% Al alloy at 425°C, but at 550°C their contributions were less significant.

In general, the tensile ductility of alloys containing 8% or more aluminum was not improved by vacuum annealing to remove hydrogen.

Of the alloys having better than 10% tensile elongation as heat treated to a stable condition, the 7% Al-3% Mo alloy showed the best creep and rupture strength properties. Also, specimens of this alloy were found to be ductile after exposure to elevated temperature creep conditions for 1000 hours. Specimens of the 6% Al-2% V and 4% V alloys were also ductile after exposure to creep conditions for 1000 hours. The 6% Al-0.5% Si and 1% Si alloys were found to embrittle upon exposure to creep conditions.

The three alloys 6% Al-0.5% Si, 6% Al-4% V and 7% Al-3% Mo were readily rolled. Tensile and bend tests on weldments of these alloys were somewhat disappointing. However, the results indicated that further study of the factors influencing ductility in unwelded and welded sheet should prove fruitful.

WADC TR 54-278 Part 2

July 1955

SUBJECT: TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATION  
INVESTIGATOR: W. F. Carew, F. A. Crossley, D. J. McPherson  
CONTRACT: AF 33(038)-22806  
CONTRACTOR: Armour Research Foundation of Illinois Institute of Technology  
ABSTRACT: The investigations directed toward development of superior titanium-base alloys for elevated temperature applications during the contract period June 1, 1954 to December 31, 1954 are reported.

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Testing was completed on a series of binary and ternary alloys not completely evaluated during the previous contract period. None of these alloys provided combinations of properties (creep and creep-rupture, room temperature ductility, and stability upon stress-aging) which were superior to previously developed alloys, particularly Ti-7% Al-3% Mo.

Room and elevated temperature tensile, creep-rupture and stability tests were made for a number of Ti-Al and Ti-Al-Mo alloys containing B, Be or C as dispersion or precipitation strengtheners and for the new ternary compositions Ti-5% Al-5% Mo, Ti-6% Al-4% Mo and Ti-6% Al-6% Mo. Again, none of the alloys demonstrated better combinations of strength, ductility and stability than Ti-7% Al-3% Mo. Boron was apparently added in insufficient quantity (0.04%) because, while ductility and stability were unaffected, negligible strengthening was obtained. Beryllium additions were apparently too high (0.5%) and caused embrittlement.

Alloys containing 5% Al-5% Mo, 6% Al-4% Mo and 7% Al-3% Mo were quite successfully fabricated to 0.060 in. sheet. Good bend properties appeared to depend upon pickling prior to heat treatment. Tensile properties for the three sheet alloys in both the welded and unwelded states are tabulated.

Variations in the previously recommended heat treatment for the Ti-7% Al-3% Mo alloy were studied with the aim of increasing commercial practicality. Solution treating must be accomplished at 800°C or higher to maintain adequate ductility. Four hours are adequate at 850°C, but longer periods of time are necessary as the temperature is lowered.

The notched specimen fatigue endurance limit of Ti-7% Al-3% Mo ( $K_t = 1.8$ ) was determined to be 43,000-45,000 psi.

Studies of the effects of sponge quality, alloy content variations, and forging practice on the properties of Ti-7% Al-3% Mo were made. Increasing the sponge hardness incrementally from 103 to 180 BHN resulted in a steady decrease of room temperature ductility in the alloy. Varying the alloy contents by  $\pm 1\%$  of each addition appeared to have little effect upon the creep and creep-rupture properties of Ti-7% Al-3% Mo. Forging practice proved to be extremely important in controlling the alloy's tensile properties. Specimens were forged initially at 2000°F to varying degrees of reduction and finished in the field at 1650° or 1800°F. There was an excellent correlation between the amount of reduction at the finishing temperatures and the tensile reduction in area of heat treated specimens. However, there was no correlation of microstructures with ductility. A need to consider the entire thermal history was indicated.

WADC TR 54-305 Part 2

September 1955

SUBJECT: HANDBOOK ON TITANIUM  
INVESTIGATOR: Heinrich K. Adenstedt  
CONTRACT: AF 33(616)-2222  
CONTRACTOR: Avco Manufacturing Corporation  
ABSTRACT: Information has been collected on specific properties of titanium and procedures for the treatment and utilization of titanium and its alloys by

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literature survey and by personal contact with the proper persons and institutions. The data have been evaluated and are condensed in this report. The two major sections of titanium technology covered are: laboratory procedures and fabrication.

WADC TR 54-546

February 1956

**SUBJECT:** PILOT PRODUCTION OF PROMISING ELEVATED TEMPERATURE TITANIUM-BASE ALLOYS  
**INVESTIGATOR:** Donald J. McPherson  
**CONTRACT:** AF 33(616)-2060  
**CONTRACTOR:** Armour Research Foundation of Illinois Institute of Technology  
**ABSTRACT:** Melting procedures and equipment to produce ingots weighing up to 100 pounds of promising titanium-base alloys developed under Air Force Contract No. AF 33(038)-22806 are described. In order to furnish ingots of this size, it was necessary to expand the existing arc melting facilities substantially. A non-consumable electrode arc furnace of 20-pound capacity employing a magnetic field to direct the arc was designed and fabricated. The capacity of the consumable electrode furnace previously developed at Armour Research Foundation was increased to a theoretical maximum of 300 pounds of titanium by the incorporation of a continuous casting mechanism and increasing the diameter of the mold. Ingots weighing up to 113 pounds and up to 6 inches in diameter were successfully cast. Titanium-base alloy stock of the following compositions was supplied to the Air Force's contractors: (1) 6% aluminum, (2) 6% aluminum-4% vanadium, (3) 6% aluminum-0.5% silicon, and (4) 7% aluminum-3% molybdenum. It was recommended that the 6% Al-0.5 Si alloy be dropped from the program since it proved to embrittle upon exposure to elevated temperature creep conditions.

Evaluation reports were received from Pratt & Whitney Aircraft Division of United Aircraft, Wright Aeronautical Division of Curtiss-Wright and the General Electric Company, Aircraft Gas Turbine Division. These reports cover only the 6% Al alloy. In general, the 6% Al alloy was found to possess good creep resistance, good weld bend characteristics and satisfactory fatigue properties. With a load of 40,000 psi at 800°F for 300 hours, a creep extension 0.08% was obtained. An as-welded bend ductility of 3.5 T and endurance limits of 55,000 and 60,000 psi were found. The alloy meets several of the requirements originally listed as desirable for titanium-base alloys early in the program.

WADC TR 54-547

December 1955

**SUBJECT:** EVALUATION OF HIGH STRENGTH WELDABLE TITANIUM BASE ALLOYS  
**INVESTIGATOR:** C. Robert Lillie  
**CONTRACT:** AF 33(616)-2321  
**CONTRACTOR:** Armour Research Foundation of Illinois Institute of Technology  
**ABSTRACT:** Six alloy compositions were investigated to determine their suitability as high strength weldable sheet to be used at elevated temperature. These compositions were: Ti-4 Al-2 V, Ti-4 Al-4 V, Ti-6 Al-2 V, Ti-6 Al-4 V, Ti-7 Al-4 V and Ti-6 Al. Tensile tests were performed on these alloys, after suitable heat treatment, both at room temperature and at 700° and 900°F, and in both welded and unwelded conditions. Bend tests were employed as a measure

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of the formability of the material. All bend tests were carried out at room temperature on heat treated material in both welded and unwelded conditions. Metallographic structure of the alloys after various heat treatments was correlated with the mechanical properties.

All of the Ti-Al-V alloys were found to provide easily rollable sheet material capable of being heat treated to a strength level competitive with stainless steel at elevated temperatures, on a strength-weight basis. The alloys were weldable, producing ductile welds which required no post-welding heat treatment. By proper heat treatment, a low ratio of yield strength to ultimate strength was obtained, indicative of good formability; subsequent aging raised the strength to a high level.

Good properties were retained in these alloys when prepared of commercial quality sponge and when subjected to commercial heat treatments.

The optimum compositions were found to lie in the composition range of Ti-4 Al-4 V to Ti-6 Al-4 V.

WADC TR 55-5

May 1955

SUBJECT: TENSILE PROPERTIES AND RHEOTROPIC BEHAVIOR OF TITANIUM ALLOYS AND MOLYBDENUM  
INVESTIGATOR: E. J. Rippling  
CONTRACT: AF 33(616)-2223  
CONTRACTOR: Case Institute of Technology  
ABSTRACT: The unnotched and notched tensile properties are described as a function of testing temperature for a series of titanium-nitrogen, and titanium-manganese binary alloys as well as for the commercial alloy, Ti 140A, and the experimental 3 Mn-complex alloy.

It was shown that the nitrogen embrittles alpha titanium by elevating its transition temperature. This brittleness, can be partially eliminated by taking advantage of a rheotropic recovery.

The Ti 140A alloy in the "as-received" condition was high in hydrogen so that a brief investigation of the effect of hydrogen in this alloy was also conducted.

Recrystallization embrittlement in commercial unalloyed molybdenum was found to be a manifestation of rheotropic brittleness.

WADC TR 55-147

November 1955

SUBJECT: INCREASING THE RATIO OF MODULUS OF ELASTICITY TO THE DENSITY OF TITANIUM ALLOYS  
INVESTIGATOR: William H. Graft, William Rostoker, David W. Levinson,  
CONTRACT: AF 33(616)-2355  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: Titanium and its alloys, although possessing many attractive properties, exhibit a somewhat lower elastic moduli to density ratio than other

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competitive engineering metals. It was the purpose of this investigation to examine alloy additions and structural conditions which might introduce improvement in the elastic modulus to density ratio.

Elastic modulus values were measured by means of a dynamic method employing electrostatic excitation, and detection. The effects of variables on the elastic properties were examined at room temperature, 500° and 750°F, and the results evaluated using the elastic modulus (E) and the elastic modulus to density ratio ( $E/\rho$ ) as the definitive criteria.

Unalloyed titanium was found to exhibit an E of  $16.2 \times 10^6$  psi for the magnesium reduced variety, and a slightly higher value for the iodide type.

The alpha stabilizing elements oxygen and nitrogen were found to affect the E and  $E/\rho$  ratio but slightly at low concentrations. The addition of aluminum, up to 8%, was found to result in marked improvement in E and  $E/\rho$  ratio.

The presence of intermediate phases on the E and  $E/\rho$  ratio was quite beneficial in some cases. TiC and TiB in their respective binary systems resulted in greatly improved elastic properties. Intermediate phases  $Ti_5Si_3$  and TiBe in their respective binary alloys exhibited only a minor effect.  $Ti_2Cu$  produced by eutectoidal decomposition in Ti-Cu alloys up to 11.2% Cu produced no significant increase in E or  $E/\rho$ .

A survey of the E and  $E/\rho$  ratio for various crystallographic directions in the base plane of  $\alpha$ -Ti in a state of preferred orientation was made. The results showed that E and  $E/\rho$  were essentially constant for various directions, and the value of E,  $15.2 \times 10^6$  psi, was somewhat lower than that for randomly oriented material.

The effect of heat treatment was found to influence the elastic modulus simply in terms of the ratios of proportions of alpha and beta.

WADC TR 55-164

January 1956

SUBJECT: A STUDY OF THE TENSILE AND CREEP- RUPTURE PROPERTIES OF FIFTEEN HEATS OF C-110M TITANIUM ALLOY SHEET

INVESTIGATOR: Frank J. Gillig  
Glen J. Guarnieri

CONTRACT: AF 33(616)-2342

CONTRACTOR: Cornell Aeronautical Laboratory, Inc.

ABSTRACT: Fifteen heats of titanium alloy RC-130-A (C110-M) have been sampled and tested at room and elevated temperatures. These data are analyzed for reproducibility and relationships between the room temperature and high temperature properties at 500°F and 700°F. The results indicate that a correlation exists between the room temperature strength properties and the yield and ultimate strengths at 500 and 700°F. However, the creep and rupture properties appear to be independent of the short time tensile strength results even at 700°F, which was the temperature used for creep testing.

WADC TR 53-373 Sup 3



SUBJECT: THE EFFECT OF GRAIN SIZE AND STRUCTURAL VARIABLES ON THE STABILIZATION OF TITANIUM ALLOYS

INVESTIGATOR: Frank C. Holden, Horance R. Ogden, Robert I. Jaffee

CONTRACT: AF 33(616)-412

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Studies have been made on the effects of alloy composition and heat treatment on the thermal stability of titanium alloys. Additions of molybdenum increase thermal stability of an alpha-beta alloy, whereas chromium decreases stability. Eutectoid decomposition products were observed in the microstructures of a Ti-5Cr alloy after 200-hour aging at 800 or 1000 F. Oxygen additives increase strength and lower ductility, without a pronounced effect on thermal stability.

Heat treatments to produce a thermally stable condition are most effective when the alloy has an acicular-type structure. This is most effectively accomplished by starting the stabilizing heat treatment in the beta field, although some improvement was observed when the alloy was originally worked in the beta field and stabilized in the alpha-beta.

Exposure to a stress of 25,000 psi during aging at 600 F did not affect thermal stability.

Stress-rupture tests did not indicate a strain-aging process in the conditions tested.

WADC TR 55-325 Part 1

March 1956

SUBJECT: THE EFFECTS OF INTERSTITIAL CONTAMINANTS ON THE NOTCH TENSILE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS Pt. 1 Iodide and Sponge Titanium

INVESTIGATOR: Neil J. Feola, Eugene P. Klier

CONTRACT: AF 33(616)-2281

CONTRACTOR: Syracuse University

ABSTRACT: The notch-tensile properties of sponge titanium contaminated with oxygen, nitrogen and carbon have been determined. The merit rating of sponge titanium in the notch-tensile test does not conform to that in the impact test.

In the small (0.3 inch diameter) specimen the levels of interstitial contamination in unalloyed titanium which may be expected to give a notch-strength ratio of unity at -65°F are as follows:

(Ti-base O<sub>2</sub> = 0.035%)

Carbon	0.4%
Oxygen	0.13%
Nitrogen	0.25%

Limited data for impact tension and sustained load tests are presented and discussed.

WADC TR 53-373 Sup 3

February 1956

SUBJECT: THE COLD EXTRUSION OF TITANIUM  
INVESTIGATOR: Alvin M. Sabroff, Paul D. Frost  
CONTRACT: AF 33(616)-2446  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Cold-extrusion studies were conducted on two grades of unalloyed titanium - AMS 4900 and AMS 4921 - to evaluate the effects of die design and extrusion reduction. By utilizing a fluoride coating and a conventional oil-graphite-molybdenum disulfide lubricant, forward extrusions with reductions of 40, 50, and 60 percent were successfully accomplished on 1-1/2-inch-diameter by 3-inch billets. Conical dies with included angles of 90, 120, and 150 degrees were used for each reduction. Working pressures were comparable to those required for cold extruding steel. For a given die angle, the extrusion pressure and average work of extrusion increased in an approximately linear relationship with the percentage reduction.

Metal-flow studies were conducted on split billets inscribed with grids. Deformation by compression and elongation was uniform over the cross sections of the bars, with maximum shear deformation occurring in the outer fibers. The amount of shear deformation increased as the die angle and percent reduction increased.

Work hardening during extrusion was nearly uniform within the bars, the overall hardness variation from center to surface being within 20 Bhn. Increases in strength varying from about 25 to 60 percent were produced under the various extrusion conditions. However, adequate ductility (elongation greater than 10 percent in 1 inch) was maintained.

The extruded bars had a smooth surface finish, with few traces of galling. No measurable die wear occurred, indicating the fluoride coating functioned satisfactorily. Dimensional tolerances on the extruded bars were maintained within 0.001 inch.

WADC TR 55-454 Part 1

February 1956

SUBJECT: A STUDY OF FACTORS AFFECTING THE UNIFORM ELONGATION OF TITANIUM AND TITANIUM ALLOYS  
INVESTIGATOR: Frank C. Holden, Horace R. Odgen, Robert I. Jaffee  
CONTRACT: AF 33(616)-2783  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: The limits of uniform elongation were measured for typical alpha, alpha-beta, and beta titanium alloys. Each alloy was studied in three microstructural conditions over a temperature range from -75 to 300 C.

Highest uniform elongation was obtained for commercial titanium (alpha), whereas that for the beta-quenched Ti-7.5Cr-7.5Mo alloy was low. The presence of massive alpha in the alpha-beta alloys increases their uniform elongation.

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The dependence of uniform elongation on temperature is marked particularly in the two-phase alloys. Maximum values were obtained at about 200C.

Measurements of uniform elongation from residual strain agree well with values of  $\sigma_{max}$ ; the values of the flow exponent  $n$  (from the flow equation  $\sigma = B\sigma^n$ ) are consistently higher.

WADC TR 55-458 Part 1

February 1956

SUBJECT: RELAXATION BEHAVIOR OF TITANIUM ALLOYS  
INVESTIGATOR: Ernest A. Sticha  
CONTRACT: AF 33(616)-2400  
CONTRACTOR: Crane Company  
ABSTRACT: Relaxation resistance of three titanium alloys in the temperature range 400 to 800F was determined to assist in evaluating possible use of titanium fasteners in aircraft for the purpose of saving weight. Tests performed in the Crane Company Engineering Laboratories showed A-110AT alloy to have the best resistance to relaxation, C-130AM alloy the next best and the 3Mn complex alloy the least. Differences between the alloys are greatest at 800F and almost disappear at 400F. All three alloys suffered some loss of impact resistance during testing at 700 and 800F but the cause of embrittlement was not apparent in the microstructure. Heat treatment of the alpha-beta alloys and cold working of the all-alpha alloy to higher strength properties failed to improve relaxation resistance at the higher temperature levels.

NONFERROUS - VANADIUM

PACKAGING

WADC TR 53-133 Part 2 (Corrected Abstract)

August 1955

SUBJECT: THEORETICAL INVESTIGATION OF THE MECHANISM OF TRANSFER OF MATERIALS THROUGH POLYETHYLENE  
INVESTIGATOR: Dr. Henry A. Bent, Jules Pinsky  
CONTRACT: AF 33(616)-112  
CONTRACTOR: University of Connecticut and Plax Corporation  
ABSTRACT: The effect of temperature on the permeability  $P$  has been found to be accurately represented by the two parameter equation

$$P = P_0 e^{-E_p/RT}$$

in which  $E_p$  and  $P_0$  are constants for the particular permeant polymer system. These parameters are calculated from carefully constructed  $\log P$  vs.  $1/T$  plots.

The size, shape, and polarity of the penetrant molecule are factors influencing  $E_p$  and for substances no more polar than acetone the expression

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$$E_p = 0.0348V + 0.75 V/L + 2.4 \quad H$$

reproduces the data to within about 0.5 kcal/mol. where polyethylene is the permeable film.

It is established that for four homologous series that

$$\log P_o = mE_p + b$$

where m and b are constants peculiar to each series.

Master plots for interpolating homologous materials and extrapolating for temperature changes are included.

#### PAINT REMOVER

#### PETROLEUM PRODUCTS

#### FUELS

WADC TR 52-35 Sup 4

January 1956

**SUBJECT:** RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS  
**INVESTIGATOR:** Micheal G. Zabetikis, George W. Jones, George S. Scott, Aldo L. Furno  
**CONTRACT:** AF 18(600)-151  
**CONTRACTOR:** United States Department of the Interior  
**ABSTRACT:** A summary of the theory of combustion, explosion and ignition is presented as well as the results of limit-of-flammability, limit-of-ignitibility, limit-of-propagation, ignition energy and ignition temperature tests on current Air Force fuels, fuel components and hydraulic fluids. These data were obtained in air, and carbon dioxide and nitrogen-enriched air at various simulated altitudes by the Bureau of Mines and other laboratories. Most of the experimental results were obtained by the members of the Bureau of Mines Gas Explosions Branch during the period February 1950 to June 1955. The combustibles investigated include the paraffins, aromatics, blends of these, JP-1, JP-3, and JP-4 jet fuels, 100 130 and 115/145 aviation gasolines, MIL-0-5606 hydraulic fluid and several experimental hydraulic fluids. The data are presented in graphic and tabular form.

WADC TR 55-219

January 1956

**SUBJECT:** INVESTIGATION OF THE ELECTRICAL AND PHYSICAL CHARACTERISTICS OF AIRCRAFT FUELS  
**INVESTIGATOR:** Richard E. Johnson, Floyd A. Andrews  
**CONTRACT:** AF 33(616)-2504  
**CONTRACTOR:** Minneapolis-Honeywell Regulator Company  
**ABSTRACT:** Continued use of the capacitance type of fuel-quantity gage in modern aircraft has necessitated a study to determine whether significant fuel characteristics are changing for the older fuel grades and to determine the characteristics of the newer fuel grades.

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The Minneapolis-Honeywell Aeronautical Division has completed such a study. Measurements were made of dielectric constants, densities, and dissipation factors on 144 samples of aviation fuel, grades 91/96, 100/130, 115/145, and JP-4. This report summarizes the results of the investigation and includes a statistical analysis of the data.

It is recommended that the calibration data currently specified by MIL-G-7817 be retained for use in calibrating noncompensated capacitance-type, fuel-gage systems. A new optimum response line and appropriate calibration data are recommended for use in calibrating the compensated systems.

#### HYDRAULIC FLUIDS

WADC TR 54-532 Part 2

January 1956

SUBJECT: HIGH TEMPERATURE HYDRAULIC FLUIDS Pt. 2 Development of Base Stock  
INVESTIGATOR: Edward S. Blake, William C. Hammann, James W. Edwards, Thomas Reichard  
CONTRACT: AF 33(616)-2623  
CONTRACTOR: Monsanto Chemical Company  
ABSTRACT: Fifty compounds have been screened for thermal stability.

Tert-alkylcarbinyl sebacates have been shown to be remarkably more stable (104-122°F) than n-alkyl sebacates. One new tert-alkylcarbinol, 2,2,4-trimethyl-1-pentanol, and eight new esters were synthesized. A polyarylphosphonate as a V.I. improver for thermally stable trialkylphosphate appeared to degrade on heating. A sufficient number of aromatic ethers were made and tested to reaffirm the high thermal stability of this chemical class and to indicate a high probability that the melting points of the aromatic ethers can be sufficiently lowered, without sacrifice in their thermal properties, to make them promising as base stock for high temperature functional fluids.

WADC TR 55-89 Part 1

October 1955

SUBJECT: PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS Part 1  
Monsanto Fluid, OS-45  
INVESTIGATOR: Oliver M. Ballentine, Donald C. Trop  
Harry M. Schiefer, 1st. Lt., USAF  
ABSTRACT: A physical and chemical evaluation has been conducted by the Wright Air Development Center, Directorate of Research, Materials Laboratory on an experimental high temperature hydraulic fluid, OS-45 developed by the Monsanto Chemical Company. This silicate base fluid was evaluated against proposed requirements for a -65° to 400°F hydraulic fluid for long life piloted aircraft. This fluid conformed to such proposed requirements as spontaneous ignition temperature, pour point, hydrolytic stability at 200°F and foaming tendency. The oxidation and corrosion characteristics of the fluid at 400°F were good in all respects with the exception of a high acid number signifying some oxidation of the oil. The viscosity of OS-45, 1.2 cs at 400°F, failed to meet the desired specification value of 2.5 at 400°F.

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Additional hydrolytic stability tests at 400°F have shown that the fluid will break down in the presence of very small percentages of water. This instability at high temperatures seems to be one of the major efficiency of this type of fluid. It is apparent from the mock up and high temperature test data available, that the present hydrolytic stability test at 200°F needs revision. Fluid stability testing above 400°F indicated that OS-45 has marginal resistance to both oxidation and thermal stability, especially when in the presence of metal catalysts. However this fluid is comparable to other silicate fluids evaluated under these requirements.

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November 1955

SUBJECT: PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL  
SILICATE-BASE HIGH-TEMPERATURE HYDRAULIC FLUIDS Pt. 2  
Hollingshead Fluid, No. 71852-B

INVESTIGATOR: O. M. Ballentine, D. C. Trop, H. M. Schiefer, 1/Lt. USAF

ABSTRACT: A physical and chemical laboratory evaluation has been conducted by the Materials Laboratory, Directorate of Research, Wright Air Development Center, on an experimental high temperature hydraulic fluid, 71852-B, which was developed by the Hollingshead Chemical Corporation. This silicate base fluid was evaluated against proposed requirements for a -65° to 400°F hydraulic fluid for long life piloted aircraft. This fluid conformed to proposed requirements as spontaneous ignition temperature, pour point, hydrolytic stability at 200°F, oxidation-corrosion, foaming, and viscosity-temperature requirements. The fluid however, failed to meet proposed requirements covering rubber compatibility. Insufficient fluid was available to perform the more significant 400°F hydrolytic stability test. This fluid had properties comparable to other fluids evaluated under the procedures contained in this report.

#### LUBRICANTS

WADC TR 53-83 Part 3

July 1955

SUBJECT: DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES

INVESTIGATOR: Edward A. Swakon

CONTRACT: AF 33(038)-23687

CONTRACTOR: Standard Oil Company (Indiana)

ABSTRACT: In the work directed toward the development of an aircraft grease suitable for use over the temperature range from -65 to 450°F and higher, emphasis was placed on the development and evaluation of arylurea greases made with the best available fluids. The chief criteria of laboratory evaluation were performance in the ABEC-NLGI Bearing Tester and the Navy Gear-Wear Tester. Sixty-seven bearing tests at 350°, 400°, 450°, and 500°F were run on thirty-two compositions. Studies were made of the effect on gear-wear tests of base fluid and thickener, of anti-wear additives in silicone greases, of diester-silicone and of Aroclor-silicone blends, and of composition of brass. Arylurea-silicone greases come closest to meeting the proposed temperature requirements, but have limited utility because of the inherently poor lubrication characteristics of silicone fluids. Six of seven arylureanon-silicone greases tested have out-performed a MIL-G-3278 grease in bearing performance at 350°F. The limiting component in the development of a grease with good lubricity for the temperature range from -65° to 450°F and above is a suitable oil.

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July 1955

SUBJECT: GREASE LUBRICATION OF HIGH SPEED ANTI-FRICTION BEARINGS  
INVESTIGATOR: John B. Accinelli, Charles R. Greene  
CONTRACT: AF 33(616)-2443  
CONTRACTOR: Shell Development Company

ABSTRACT: A study of the failure mechanisms of grease lubricated anti-friction bearings operating in the DN range from  $1.0 \times 10^6$  to  $2.0 \times 10^6$  was the main object of this project. All test work was conducted at the Shell Development Company using a rig designed by this company. After necessary rig development work and bearing selection tests, preliminary work on the effect of various grease and operational variables on the performance of ball bearings operating up to DN values of  $1.5 \times 10^6$  was initiated. The problem seems to be one of maintaining an effective lubricant film on the working surfaces of the bearing under the very strong stripping action of centrifugal force, high sliding velocity and very high rate of shear. The grease property apparently most critical to lubrication at high speeds is viscosity of the oil. Operation at high (300°F) temperatures also seems to aid lubrication.

February 1956

SUBJECT: GREASE LUBRICATION OF HIGH SPEED ANTI-FRICTION BEARINGS  
INVESTIGATOR: John B. Accinelli, Charles R. Greene  
CONTRACT: AF 33(616)-2443  
CONTRACTOR: Shell Development Company

ABSTRACT: The work reported is a continuation of the study of grease lubrication of ball bearings operating at very high DN values ( $1.0 \times 10^6$  to  $2.0 \times 10^6$ ). The work was performed at the Shell Development Company on two high speed rigs designed and built for the Air Force by this company. These rigs, utilizing 20 mm ball bearings, required considerable development work in order to obtain satisfactory operation at the desired DN values. Before work on greases was initiated each rig was calibrated with oil-air mist lubrication by running continuously for 100 hours at  $1.2 \times 10^6$  DN in duplicate tests with two oils, a mineral oil of SAE grade 1010 (MIL-O-6081) and a diester synthetic oil (MIL-L-6085A). The rigs are now ready for the grease studies. In the course of these calibration runs it was found that slight dynamic unbalance of the rotating components has a very deleterious effect on bearing operation, and that for successful operation, in the very high range of DN, micro balancing of the rotor is required. The very high centrifugal forces encountered in very high speed operation seem to cause continuous stripping of the lubricant film, and this action requires that lubricant be continually applied to the bearing at a fairly high rate of flow in order to obtain prolonged operation. This factor may limit the applicability of greases as lubricants for very high speed ball bearing application.

The laboratory investigation of grease properties in a force field obtained by centrifugation has been continued. It has been shown that bulk greases will not adhere to metal surfaces subjected to the unimpeded stripping action of centrifugal forces comparable to those encountered in some areas of the operating high speed bearing. Oil loss from grease structures, through the shearing and crushing action of centrifugal force, has been shown to vary with grease type and

composition. The results obtained have permitted some qualitative conclusions on the way the grease properties studied may affect bearing operation.

WADC TR 55-187

August 1955

SUBJECT: INVESTIGATION INTO THE USE OF HETEROCYCLIC COMPOUNDS AS LUBRICANT ADDITIVES

INVESTIGATOR: George B. Butler, O. Lee Gordon, Louis A. Haynes

CONTRACT: AF 33(616)-2391

CONTRACTOR: Peninsular ChemResearch, Inc.

ABSTRACT: A literature survey and synthesis program were carried out in order to prepare various nitrogen and sulfur, selenium, or oxygen-containing heterocycles for evaluation as antioxidant, anti-wear and extreme pressure additives for lubricants, hydraulic fluids and greases. In addition, a number of organoselenium compounds of the selenide and diselenide types were synthesized, primarily for evaluation as high-temperature oxidation inhibitors. Solubilities of the prepared compounds in various referenced fluids were determined.

WADC TR 55-187 Part 2

February 1956

SUBJECT: INVESTIGATION INTO THE USE OF HETEROCYCLIC COMPOUNDS AS LUBRICANT ADDITIVES

INVESTIGATOR: George B. Butler, O. Lee Gordon, Louis A. Haynes

CONTRACT: AF 33(616)-2391

CONTRACTOR: Peninsular ChemResearch, Inc.

ABSTRACT: A literature survey and synthesis program were carried out in order to prepare various nitrogen and sulfur-selenium-, or oxygen-containing heterocycles for evaluation as antioxidant, anti-wear and extreme pressure additives for lubricants, hydraulic fluids and greases. In addition, a number of organoselenium compounds of the selenide and diselenide types were synthesized, primarily for evaluation as high-temperature oxidation inhibitors. Solubilities of the prepared compounds in various referenced fluids were determined.

WADC TR 55-190

July 1955

SUBJECT: DEVELOPMENT AND EVALUATION OF A GREASE FOR -100°F to +350°F

INVESTIGATOR: Ernest W. Nelson, C. Richard Bergen, Billy P. Scott,

Warren W. Woods, Richard M. Tillman

CONTRACT: AF 33(616)-2364

CONTRACTOR: Continental Oil Company

ABSTRACT: This report describes the history and present status of the development of a general purpose grease to be operational over a -100°F. to +350°F. temperature range. Experimental work has been conducted during the past 12 months by Continental Oil Company under AF 33(616)-2364 and is to be continued under a nine-month extension contract.

These studies have been limited to development of greases thickened by colloidal calcium carbonate coated with calcium salts of aliphatic dicarboxylic acids. Using diester vehicles, it now appears probable that a balanced formulation

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can be made which will meet specifications for consistency, bleeding, work stability, dropping point, evaporation loss, low temperature torque, and water resistance. Some revision in composition for present greases will be required since incorporation of work stability and water resistance additives has been found to induce changes in consistency and bleeding which must be offset by slight changes in the thickener. In addition, an oxidation inhibitor must be incorporated prior to starting prolonged bearing tests at high temperatures.

WADC TR 55-190 Part 2

April 1956

SUBJECT: DEVELOPMENT AND EVALUATION OF A GREASE FOR -100 to +350°F  
INVESTIGATOR: Ernest W. Nelson, Warren W. Woods, C. Richard Bergen,  
William P. Scott, Richard M. Tillman

CONTRACT: AF 33(616)-2364

CONTRACTOR: Continental Oil Company

ABSTRACT: The thickener for this wide temperature range grease development has been limited to a colloidal calcium carbonate coated with the calcium salts of aliphatic dicarboxylic acids. The aliphatic diesters were studied as possible base fluids. Although promising in many respects, their use is precluded because of high evaporation rates and lack of a sufficiently active oxidation inhibitor at 350°F. Greases prepared with certain hexa alkoxy disiloxanes appear promising. Formulations using the base oil hexa (2-ethyl hexoxy) disiloxane very nearly meet or surpass target requirements, the lone exception being high temperature performance. Here a maximum of 350 hours has already been achieved. Formulation changes designed to improve performance of such greases have been developed, but testing has been hampered because of the short supply of the base fluid.

WADC TR 55-240

December 1955

SUBJECT: BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP  
LUBRICATING GREASES

INVESTIGATOR: G. J. Young, J. J. Chessick, David Chu,  
A. Uliny, E. Borger, A. C. Zettlemoyer

CONTRACT: AF 33(616)-2440

CONTRACTOR: Lehigh University

ABSTRACT: Pertinent physical properties of several oils and surface characteristics of various types of thickening agents have been determined in order to index the nature and extent of the oil-thickener interface in non-soap grease systems. These properties of the oils and thickening agents are correlated with the behavior of grease systems formulated from them. In addition, modifications of the oil-thickener interfacial region by traces of water and by polar organic additives have been studied. Ramifications of the alteration of this interfacial zone on gel structure and stability of grease systems are discussed.

WADC TR 53-373 Sup 3

SUBJECT: MICRO LUBRICANT TEST METHODS Part 2 Swelling of Synthetic Rubber  
Color of Lubricating Oil and Petrolatum Hydrolytic Stability  
INVESTIGATOR: John B. Christian, Harry M. Schiefer, 1/Lt., USAF  
ABSTRACT: This report introduces micro methods for the determination of the swelling of synthetic rubbers in aircraft greases and hydraulic oils, and the determination of color of lubricating oil and petroleum through the use of the A.S.T.M. Union Colorimeter, and the determination of hydrolytic stability of aircraft oils.

These methods were arrived at through the comparison of modified small scale tests with the full scale tests.

WADC TN 55-372

August 1955

SUBJECT: A SURVEY OF THE LITERATURE ON THE LUBRICATION OF TITANIUM  
INVESTIGATOR: Robert Benzing, 1/Lt., USAF, Ray E. Bryant, 1/Lt., USAF  
ABSTRACT: A survey of the literature on the lubrication of titanium was made. The work studied is broken down into three main groups: Frictional and Lubricating Properties, Surface Coatings and Treatments, and Machining Practices. A brief discussion summarizing the literature is given which outlines the present status of the work with suggestions for future practices.

WADC TN 55-554

January 1956

SUBJECT: INVESTIGATION OF LITHIUM DIHYDROXY AND POLYHYDROXY STEARATES  
AS GREASE THICKENERS  
INVESTIGATOR: Robert J. Benzing, 1/Lt., USAF  
ABSTRACT: A dihydroxy and a polyhydroxy stearic acid supplied by the Baker Castor Oil Company were evaluated as potential raw materials in the reproduction of thickening agents for lithium soap greases. The greases made with these soaps are highly thixotropic, but the greases made from the dihydroxy stearic acid are better in this respect than the greases made with the polyhydroxy stearic acid.

### PLASTICS, STRUCTURAL

WADC TR 52-183

November 1955

SUBJECT: ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN, PLASTICS  
FOR AIRCRAFT  
INVESTIGATOR: Donald G. Coleman  
CONTRACT: Amend A2(55-295) September 13, 1954 To  
Do (33-616) (53-20)  
CONTRACTOR: Forest Products Laboratory  
ABSTRACT: Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1955 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Much of the data obtained during the year were incorporated in the 1955 edition of ANC-17 Bulletin.

WADC TR 53-373 Sup 3

January 1956

SUBJECT: EVALUATION AND DEVELOPMENT OF PLASTIC LAMINATED BACKING BOARD MATERIALS  
INVESTIGATOR: Charles C. Surland  
CONTRACT: The United States Rubber Company  
CONTRACTOR: Sup Agreement No. S3(54-1266)  
ABSTRACT: Different types of backing board materials were tested by internal explosion techniques to examine the feasibility of duplicating gunfire damage. Pressure deflection tests on a simulated airframe installation were made to relate mechanical properties of the backing boards to their support characteristics for both self-sealing and bladder type fuel cells.

Empirical equations were developed for the deflection of backing boards under fluid pressures. The tests included encountered the maximum and minimum spans which would normally be in aircraft structures. Equations were correlated with the physical properties of the backing boards as the basis for establishing a method of specifying backing boards.

Production trials of USV CR 88 backing board were made to examine the feasibility of production by continuous methods and furnish materials to the WADC.

WADC TR 55-186

November 1955

SUBJECT: ASBESTOS FIBER REINFORCED PLASTIC LAMINATES  
INVESTIGATOR: Conrad C. Spatz  
CONTRACT: AF 33(616)-2257  
CONTRACTOR: Johns-Manville Corporation  
ABSTRACT: An investigation was started to determine the effectiveness of asbestos in reinforcing low-pressure laminating resins. This report covers the evaluation of laminates prepared from various asbestos papers and a phenolic resin.

Laminates were made that meet the MIL-P-8013 (USAF) specifications on tensile, edgewise compressive, and flexural strengths at room temperature for glass mat reinforced polyester laminates. Dry flexural strength was retained 100% after two hours boiling in water and after immersion in water for sixty days at room temperature. Room temperature flexural strength was retained close to 100% after seven days exposure to 300°F and tested at 300°F. A 65% strength loss occurred after 1/2 hour at 500°F with no further loss up to seven days exposure. The paper was found to be fairly non-directional in strength.

WADC TR 55-256

December 1956

SUBJECT: EFFECTS OF MOLDING PRESSURE ON THE STRENGTH PROPERTIES OF SEVERAL TYPES OF GLASS-FIBER REINFORCED PLASTICS  
INVESTIGATOR: Samuel D. Toner, Frank W. Reinhart, Irvin Wolock  
CONTRACT: PO AF 33(616)-53-14  
CONTRACTOR: National Bureau of Standards

WADC TR 53-373 Sup 3

**ABSTRACT:** An investigation was made to determine the effects of molding pressure on the mechanical properties of glass-fiber reinforced laminates fabricated from a polyester resin reinforced with woven glass fabric or with 2 oz. or 8 oz. unoriented glass fiber mat.

The laminates were fabricated in an open mold at molding pressures of 1, 10, and 100 lb/in<sup>2</sup>, and in a closed mold at 10, 100, and 500 lb/in<sup>2</sup>. The resin content of the free-edge panels varied with the molding pressure, while that of the closed-edge panels was maintained at a constant value for each type of reinforcement.

Flexural, tensile and compressive strengths and flexural and tensile moduli of elasticity were determined for dry specimens as well as for specimens that had been immersed in water at 73.5°F (23°C) for thirty days. Specific gravity, resin content, and voids content were also determined.

The test results indicate that the properties of laminates fabricated in the closed mold do not vary appreciably with changes in molding pressure. However, the flexural and tensile properties of panels molded in the open mold increase as the pressure increases from 1 to 10 lb/in<sup>2</sup> but do not change appreciably at higher molding pressure. Compressive strength shows a tendency to decrease with increasing molding pressure.

The results obtained for the closed-mold panels were in close agreement with those of the open-mold panels having the same reinforcement and similar resin content.

WADC TR 55-290

November 1955

**SUBJECT:** THE DEVELOPMENT OF FIBROUS GLASSES HAVING HIGH ELASTIC MODULI  
**INVESTIGATOR:** George R. Machlan  
**CONTRACT:** AF 33(616)-2422  
**CONTRACTOR:** Owens-Corning Fiberglas Corporation  
**ABSTRACT:** A fifty percent increase in modulus of elasticity of fibrous glass was achieved by the continuous formation of fibers of a calcium aluminate glass in a small textile glass bushing.

The resistance of these glasses to chemical attack by water and water vapor is much less than that of commercially produced textile fibrous glass. The glasses are resistant to hydrofluoric acid but are completely soluble in hydrochloric acid.

The dielectric constants of these glasses were found to be higher than the dielectric constant of present textile glass and the loss tangents were found to be approximately the same as that of present textile glass.

Several compositions were investigated which contained tin oxide as the major constituent but no glasses were found.

An exploratory study of glass-plastic combinations from the calcium aluminate glass was initiated. Volan A was found to be the best coupling agent tested for this glass.

WADC TR 53-373 Sup 3

SUBJECT: THE RAIN EROSION OF AIRCRAFT MATERIALS  
INVESTIGATOR: R. R. Lapp, Raymond H. Stutzman, Norman E. Wahl  
CONTRACT: AF 33(600)-6469  
CONTRACTOR: Cornell Aeronautical Laboratory  
ABSTRACT: Many engineering problems have been encountered with the advent of subsonic and supersonic aircraft. One of these problems is the phenomenon of erosion during flight through rain of coatings, structural plastic, and metal parts on the exterior of high speed aircraft.

This report is a compilation of data on all the metallic and non-metallic aircraft materials tested from May 1947, to August 1955, using the rotating arm erosion apparatus. Most of the tests were conducted at 500 mph in 1 in/hr rainfall; however, the early tests were conducted at speeds as low as 150 mph.

The materials which were evaluated include plastics, both thermoplastic and thermosetting types, elastomeric materials, lacquers and enamels, inorganic materials such as glass and ceramics, and metals.

This report summarizes factors affecting the rain erosion resistance of materials such as velocity, airfoil radius, coating support, the influence of angle of impact, type of core material and surface defects.

The data compiled here are referenced to the specific report in which the tests are described in detail at the time they were conducted.

The purpose of this report is to evaluate the performance of a number of products for a specific application. Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

#### POLYMERS AND SYNTHESIS STUDIES

WADC TR 54-102 Part 2

February 1956

SUBJECT: ORGANOSILOXANE POLYMERS CONTAINING POLAR GROUPS IN THE SIDE CHAINS  
INVESTIGATOR: Victor D. Aftandilian, Eugene G. Rochow  
CONTRACT: AF 33(616)-479  
CONTRACTOR: Harvard University  
ABSTRACT: In accordance with the proposed objectives of this research a study of the synthesis of polymerizable organosilicon monomers containing organoboron groups was made. Such monomers are intended to be used in conjunction with nitrogen-containing monomers to increase inter-chain attraction in the polymer

WADC TR 53-373 Sup 3

A high-vacuum system was built and by this means large quantities of dimethylboron bromide were prepared.

Side-chain chlorination of methylsilanes were carried out by photochemical reactions. In particular, methyl(chloromethyl)dichlorosilane was prepared by this method and was converted to methyl(chloromethyl)bis(o-cresoxy) silane, a hitherto unknown compound.

A method was found to convert chlorosilanes to silanes, using lithium aluminum hydride, without reducing chloromethyl groups attached to silicon. Methyl(chloromethyl) silane was prepared for the first time by this process.

Attempts to prepare a Grignard reagent from methyl(chloromethyl) bis(o-cresoxy)silane failed, probably because of steric hindrance of the two bulky o-cresoxy groups. However, by replacing the o-cresoxy groups with hydrogen atoms the preparation of the Grignard reagent was successful.

The reaction of dimethylboron bromide with the Grignard reagent of methyl(chloromethyl) silane appeared to proceed satisfactorily. The product was separated as an adduct of dimethylamine. Many attempts were made to find a suitable solvent for purification of this compound. Although the product contained boron, silicon, hydrogen, and nitrogen, the results of the analysis indicate presence of impurities. The synthesis of polymerizable silane monomers containing organoboron groups thus appears feasible by the methods developed under this project, but further improvements in separation are necessary in order readily to prepare considerable amounts of polymer for evaluation.

WADC TR 55-25

September 1955

SUBJECT: POLYMERS DERIVED FROM DIHYDROPERFLUOROBUTYL ACRYLATE  
INVESTIGATOR: Fred W. Knobloch, Horace C. Hamlin, Maj., USAF  
ABSTRACT: Exploratory copolymerizations of 1,1-dihydroperfluorobutyl acrylate with various other monomers selected from such general classes as 1,3 dienes, unsaturated acids, acrylamides, haloacrylates, vinyl alkyl ethers, vinyl silanes and allyl esters of unsaturated acids are described. Preliminary compounding recipes and evaluation data are presented for those systems which appeared to be of definite interest in connection with the development of specialty elastomers for Air Force applications. Attempts to prepare a copolymer possessing a balance of properties superior to that which exists in poly 1,1-dihydroperfluorobutyl acrylate were successful. However, considerable information was obtained regarding the behavior of 1,1-dihydroperfluorobutyl acrylate in copolymerization reactions and the search for potentially valuable polymers based on this important new monomer was extended to include preliminary investigation of many copolymer compositions not previously studied.

WADC TR 55-26 Part 1

March 1955

SUBJECT: RESEARCH ON BORON POLYMERS LITERATURE SURVEY  
INVESTIGATOR: William L. Ruigh, Charles E. Erickson  
CONTRACT: AF 33(616)-2057  
CONTRACTOR: Rutgers University

WADC TR 53-373 Sup 3

**ABSTRACT:** A literature search has been made in the field of boron polymers. Polymers derived from boric acid esters are all sensitive to hydrolysis by water as are borazole and its derivatives. Boronamides and N-boroureas have been claimed to yield strong fibre and film-forming polymers.

The role of boron compounds in the technology of silicone and other synthetic rubbers is important. The patent literature does not satisfactorily reveal the chemical functions played by boron compounds.

Research has been initiated on the preparation of polymeric boronamides derived from stabilized boronic acids and bifunctional isocyanates.

WADC TR 55-26 Part 2

May 1955

**SUBJECT:**

**INVESTIGATOR:** William L. Ruigh, Frank Gunderloy, Charles E. Erickson, Michael Sedlak

**CONTRACT:** AF 33(616)-2057

**CONTRACTOR:** Rutgers University

**ABSTRACT:** An exploratory study has been made of certain boron compounds and polymers in connection with the development of plastics and elastomers, which are thermally stable as well as oil and fuel resistant.

The stable polymeric "boronamides" derived from boronic acids and bi-functional isocyanates and claimed in a recent patent have been shown to be essentially polyureas admixed with soluble boron compounds.

Esters and polymers based on tricovalent boron are hydrolytically unstable and therefore this project was directed toward the synthesis of quadri-covalent chelate and quasi-chelate boronic acids and borates. The diethanolamine ester of benzene-boronic acid has been prepared. It was stable to hydrolysis, and could be recrystallized from water. Allylboronic acid and its esters have been synthesized and studied primarily as intermediates for *o*-dimethylaminopropane boronic acid and related compounds which as yet have not been prepared. This work is being continued.

WADC TR 55-193 Part 2

February 1956

**SUBJECT:** FLUORINE-CONTAINING POLYETHERS

**INVESTIGATOR:** Ogden R. Pierce, Donald D. Smith, Robert M. Murch

**CONTRACT:** AF 33(616)-2417

**CONTRACTOR:** Dow Corning Corporation

**ABSTRACT:** This research was initiated to explore the preparation of flourine-containing polyethers for possible use as thermally stable elastomers.

Investigations of several moners were conducted. Indications are that hypohalogenation of flourine containing olefins generally is unsuccessful.

The preparation of epoxides by reaction of flourine containing olefins with peracids has been briefly explored.

WADC TR 53-373 Sup 3

The synthesis of epoxides using flourine containing ketones as precursors was studied and the results indicate the approach warrants exploitation.

In the area of polymerization the use of free radical initiators was studied. In no instance was a satisfactory rubber obtained. The polymers prepared were quite solvent resistant and could conceivably find applications where good rubbery character is not necessary.

WADC TR 55-461

April 1956

SUBJECT: GAMMA RAY INDUCED ADDITION OF BROMOTRICHLOROMETHANE TO OLEFINS  
INVESTIGATOR: A. M. Lovelace, 1/Lt. USAF, D. A. Rausch, 1/Lt. USAF  
ABSTRACT: A new technique in organic synthesis has been applied to the preparation of intermediates for monomers to be used in the polymerization of new useful fluid plastic and elastomeric material for high temperature applications in military aircraft. The addition of bromotrichloromethane to olefins has been effected with gamma radiation. The well-known free radical addition of bromotrichloromethane to olefins has been studied utilizing Cobalt<sup>60</sup> as the energy source for the generation of free radicals. It has been found that additional reactions can be conducted in this manner resulting in good yields of the simple one to one addition products. No unexpected side reactions have been encountered and the adducts of bromotrichloromethane with 1-octene (85%), allyl chloride (51%), vinylacetate, (70%), cyclohexene (65%) and vinyltrichlorosilane (47%) have been obtained in the yields shown after radiation dosage of  $2.25 \times 10^5$  roentgens (30 minutes exposure). In all cases the resulting products were shown to be identical with those reported by other investigators using organic peroxides or ultra-violet light.

Using the system of bromotrichloromethane and 1-octene a study of the dependency of yield on radiation dosage was carried out. The variation in molar ratio of the reactants was shown to give a good yield (74%) of 1,1,1-trichloro-3-bromononane even when an equi-molar mixture of the methane and olefin was used.

The application to other reactions is being investigated.

WADC TR 55-146

June 1955

SUBJECT: ORGANIC DERIVATIVES OF GERMANIUM ORTHO ESTERS OF 2-ALKOXYETHANOLS  
INVESTIGATOR: Harold Rosenberg, Donal F. Kippax,  
Elizabeth J. Bartholomew  
ABSTRACT: An investigation was conducted to determine the applicability of germanium ortho esters, similar to the organosilicates, as base stock materials for high-temperature fluids and lubricants. A series of tetrakis(2-alkoxyethoxy) germanes was synthesized by the reaction of sodium 2-alkoxyethoxides with germanium tetrachloride after it was found that the germanium halide, unlike its silicon analog, failed to react with a 2-alkoxyethanol. Certain of the physical properties of the germanium ortho esters and their resemblance to those of the corresponding



silicon derivatives are discussed. In the course of this work a new glycol ether, 2-cyclohexyloxyethanol, was prepared by the action of ethylene chlorohydrin on the sodium salt of cyclohexanol in the presence of xylene.

WADC TR 54-613 Part 1

July 1955

SUBJECT: ORGANOSILANES AND RELATED COMPOUNDS AS HIGH-TEMPERATURE LUBRICANTS  
Part 1 Synthesis and Properties of Dodecyltrialkylsilanes

INVESTIGATOR: Harold Rosenberg, Elizabeth J. Bartholomew,  
James D. Groves, 1/Lt., USAF

ABSTRACT: A series of studies was conducted to uncover new types of organosilicon compounds with suitable properties for extreme-temperature lubricant and hydraulic fluid applications. An introduction to these investigations in the form of a study of the synthesis and properties of one class of unsymmetrical tetraalkylsilanes is presented. A series of dodecyltrialkylsilanes was prepared and certain of the physical properties of these compounds correlated with molecular structure. These materials were found to be liquid over a wide temperature range and, in certain cases, to possess extremely high boiling points. On the basis of preliminary evaluation, these compounds appear to have promise as base stocks for certain extreme-temperature fluid and lubricant applications.

WADC TR 55-221

August 1955

SUBJECT: INVESTIGATION OF CONDENSATION TYPE ELASTOMERS

INVESTIGATOR: George C. Schweiker, Rudolph N. Delco,  
Russell R. White

CONTRACT: AF 33(616)-2421

CONTRACTOR: Hooker Electrochemical Company

ABSTRACT: The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. High thermal stability and resistance to fuels, synthetic oils and hydraulic fluids are major requirements for such elastomers.

Various fluorine- and sulfur-containing difunctional compounds have been prepared and high molecular weight polyesters have been synthesized. The linear polyesters made from fluorine-containing glycols exhibit rubber-like properties and are not brittle at low temperatures, in some cases. When fluorine is contained in these polyesters in adequate amounts, they are resistant to hydrocarbon solvents.

Preliminary cross-linking experiments have given a tough elastomer from hexafluoropentylene adipate, without added reinforcing fillers.

The materials described are the first examples of fluorine-containing polycondensation elastomers and appear to be of considerable interest.

WADC TR 55-220

August 1955

SUBJECT: DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS

INVESTIGATOR: Paul Tarrant

CONTRACT: AF 33(600)-26593

CONTRACTOR: Peninsular ChemResearch, Inc.

WADC TR 53-373 Sup 3

**ABSTRACT:** The preparation of a homopolymer from 3, 3, 3-trifluoropropyl-methyldichlorosilane has been accomplished on a small scale.

Experimental conditions have been studied for the reaction of trichlorosilane and methyldichlorosilane with olefins. For hydrocarbon olefins, the best yield of addition product was obtained at 250°C without a peroxide catalyst. The use of a platinum catalyst supported on charcoal allows the use of lower reaction temperatures and shorter reaction times in these additions.

The additions of silanes to a number of fluoroolefins have been carried out. Both methyldichlorosilane and trichlorosilane have been employed as addenda and  $\text{CF}_2=\text{CFCl}$ ,  $\text{CH}_2=\text{CF}_2$ ,  $\text{CHF}=\text{CF}_2$ ,  $\text{CF}_2=\text{CFCF}_3$ ,  $\text{CH}_2=\text{CHCF}_3$ ,  $\text{CH}_2=\text{CHC}_3\text{F}_7$  and  $\text{CH}_2=\text{C}(\text{CF}_3)\text{CH}_3$  as the fluoroolefins. The best yield by far was obtained using 3,3,3-trifluoropropene which gave a 72% yield of adduct,  $\text{CF}_3\text{CH}_2\text{CH}_2\text{SiCl}_3$ , with trichlorosilane. The physical properties of the various compounds have been determined.

Vinylsilanes have been found to react with molecules such as  $\text{CCl}_3\text{Br}$ ,  $\text{CF}_2\text{Br}_2$ ,  $\text{CF}_3\text{I}$ ,  $\text{CF}_3\text{Br}$ ,  $\text{CF}_3\text{BrCFClBr}$  and  $\text{CF}_2\text{ClCFClI}$  to give compounds containing halogen atoms on an alkyl side chain. The properties of these compounds have been studied.

WADC TR 54-599

August 1955

**SUBJECT:** ELASTOMERIC POLYPHOSPHATES

**INVESTIGATOR:** R. A. Hubbard, II

U. P. Strauss

**CONTRACT:** AF 33(616)-2059

**CONTRACTOR:** Rutgers University

**ABSTRACT:** The research on "Elastomeric Polyphosphates" covered in this report was carried out by R. A. Hubbard, II, and U. P. Strauss at Rutgers University from 1 July 1953 to November 1954. The problem is the preparation of an elastomer based on the high molecular weight polyphosphate chain. Since the known polyphosphates are essentially linear polyelectrolytes occurring in the glassy or crystalline form, the problem consists of two parts. First, the strong interionic forces which cause stiffness of the materials must be overcome; second, the chains must be cross-linked. The first of these problems is attacked by the use of large cations and plasticizers, the second by the use of polyvalent cations; possible chemical methods of tying the chains together are discussed. Some elastomeric products have been obtained, showing the essential soundness of this approach. However, none of the products prepared so far have the required thermal stability, even though the polymeric chain is stable. As a result of this research, we envision the need of a thermally stable cation with a built in plasticizer.

#### RUBBER

WADC TR 54-190 Part 3

December 1955

**SUBJECT:** DEVELOPMENT OF HIGH-TEMPERATURE OIL-RESISTANT RUBBER

**INVESTIGATOR:** William J. Mueller,

Richard A. Clark,

Louis E. Novy,

Randall G. Heiligmann

**CONTRACT:** AF 33(616)-476

**CONTRACTOR:** Battelle Memorial Institute

WADC TR 53-373 Sup 3

**ABSTRACT:** This report describes research to develop two types of rubber composition. These were (1) a composition that will withstand 500 to 1000 hours of immersion in diester-type lubricating oil (Turbo Oil-15) at 350 to 400 F, and (2) a composition that will withstand immersion for the same time period in silicate ester-type hydraulic fluids (OS-45 or MLO-8200) at 400 F.

The best compositions for use in hydraulic fluids for an aging time of 168 hours at 400 F were prepared from Neoprene WRT. For this time period, these compositions met nearly all of the WADC minimum target specifications. For longer aging periods, stocks made with Acrylon BA-12 and with Philprene VP-A appeared to be the most promising, although only limited work has been done with these polymers. Hycar 1014 and Philprene VP-25 were less promising polymers than Neoprene WRT, and Hycar 1072 and butyl rubber were completely unsuitable.

Compositions to be used in Turbo Oil-15 were evaluated at both 350 F and 400 F. At 350 F, poly-FBA compositions were able to meet all of the minimum target specifications of this project, and at 400 F they failed only because of low tensile strength after aging (300 to 350 psi, compared with a minimum target of 800 psi). At 350 F, Hycar 4021 stocks missed the minimum target requirements because they swelled about 6 per cent above the 30 per cent maximum. At 400 F, Hycar 4021 stocks had about 25 per cent excessive swell, fell down in stress-strain properties, and cracked after 500 hours of aging. Further efforts to overcome the tendency of Acrylon EA-5 stocks to crack, even when aged at 350 F, were not successful.

WADC TR 55-351

January 1956

**SUBJECT:** TESTING OF SILICONE RUBBER AT ELEVATED TEMPERATURES  
**INVESTIGATOR:** Aldo J. DeFrancesco  
**CONTRACT:** AF 33(616)-2542  
**CONTRACTOR:** The Connecticut Hard Rubber Company  
**ABSTRACT:** An apparatus to measure tensile strength, elongation, and tear strength of silicone rubber at elevated temperatures was designed and constructed. Fifteen different silicone compounds were tested at room temperature, 212°F and 400°F.

Although it is known that physical properties of silicone rubber are only slightly affected after the rubber is aged at high temperatures, it was found that they are greatly reduced when measured at high temperatures.

The best tensile strength, elongation and tear strength at 400°F were displayed by Cohrlastic HT Heat Stable (CHR 5908A), a compound containing Valron (DuPont) as the filler. CHR Compound 5901, composed of Dow Corning 6-128 with glass wool as an additional filler, also exhibited good tensile and tear strengths at 400°F.

The least reduction in these properties at 400°F, as compared with results obtained on the silicone compounds at room temperature, was displayed by CHR 5901. Dow Corning Silastic 152, postcured at 600°F, showed the least change in elongation.

WADC TR 53-373 Sup 3

Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

WADC TR 55-377

October 1955

SUBJECT: A STUDY AND EVALUATION OF KEL-F ELASTOMER

INVESTIGATOR: R. E. Headrick

ABSTRACT: The bulk of the work reported herein is a summary of experimental compounding while trying to compound for low compression set and good chemical resistance.

Initial compounding of Kel-F Elastomer revealed the most promising compounds for low compression set and chemical resistance were those cured with benzoyl peroxide. The compound having the lowest set in this effort was 266-62-1. This compound has a set of 40 percent when compressed 30 percent at 250°F for 70 hours.

Immersion tests in experimental hydraulic fluids composed of silicate esters or "silicone oils" indicate that Kel-F Elastomer may prove useful for aircraft hydraulic system applications up to 400°F. Tests also indicate that this elastomer when properly compounded has exceptional resistance to potential rocket fuels such as fuming nitric acid and may prove useful for hose, seals, protective clothing and other items for contact with these fluids.

Of particular importance to the rubber compounder is the discovery that prolonged milling is required during compounding to obtain uniform physical properties from the compounded elastomer. Variation in milling in time can change final physical properties of the cured elastomer as much as 100 percent, as least on the polymer produced to date.

WADC TR 55-381

November 1955

SUBJECT: POLY FBA - A FLUORINATED ACRYLIC ELASTOMER FOR HIGH TEMPERATURE SERVICE IN THE PRESENCE OF AIRCRAFT FUELS AND LUBRICANTS

INVESTIGATOR: Horace C. Hamlin, Maj. USAF

ABSTRACT: A large number of fluorine containing polymeric systems have been investigated by the Minnesota Mining & Manufacturing Company under Air Force Contract No. 33(038)-515. One of the most successful developments, poly 1,1 dihydroperfluorobutyl acrylate (poly FBA) exhibits good rubbery characteristics, excellent resistance to many fuels, lubricants, solvents, chemicals, and ozone, plus very good stability at elevated temperatures. Compounding and processing studies have been made, and tests conducted in various media at temperatures up to 550°F. Results of laboratory and simulated service tests show poly FBA to be vulcanizable through the action of certain metal oxides or organic amines, reinforcing with carbon blacks and some inorganic fillers, and readily handled on standard rubber processing equipment. Tensile strength ranges from 1000 to 1400 pounds and elongation from 200 to 400%. It is highly resistant to the effects of hydrocarbon fuels, even at elevated temperatures, and has been successfully tested in the presence of synthetic lubricants for up to 300 hours at 400°F, and for shorter periods at higher temperatures.

WADC TR 53-373 Sup 3

Poly FBA is also highly resistant to the chemical action of fuming nitric acid, although quite permeable to it. It is completely resistant to ozone. Low temperature properties are limited to about 0°F unplasticized, although it retains good elasticity almost down to the brittle point.

WADC TR 55-439

February 1956

SUBJECT: REINFORCEMENT OF SILICONE RUBBER WITH CARBON BLACK

INVESTIGATOR: Aldo J. DeFrancesco

CONTRACT: AF 33(616)-2542

CONTRACTOR: The Connecticut Hard Rubber Company

ABSTRACT: The silicone polymer, Linde W-96, when filled with various carbon blacks, was successfully cured to form a firm, well-reinforced rubber. Not all carbon blacks, however, are satisfactory. In general, an optimum loading of most acetylene and furnace blacks can be used. Specifically, Shawinigan, Philblack O, Philblack A, Pelletex and Thermax produced good stocks. Reinforcement approached that obtained with many white fillers in silicone rubbers. All of the stocks containing the above fillers showed good aging characteristics at 400° and 450°F.

Channel blacks severely retarded the cure, and efforts were made to prevent this effect. A cure was obtained with di-tertiary-butyl peroxide in the case of a compound comprising a blend of Linde W-96 and a methyl siloxane, containing Spheron 6 as the filler. This cure was accomplished by allowing the stock to cool to room temperature before pressure was removed from the mold.

Preheating Linde W-96: carbon black masterbatches, as suggested by the Kilbourne-Davis patent, U. S. 2,709,161, did not improve the reinforcement qualities of the carbon blacks. Benzoyl peroxide was successfully used as a curing agent when a stock containing Shawinigan Black was heat-treated for 16 hours at 480°F.

#### SANDWICH CONSTRUCTION

WADC TR 52-184 Sup 3

November 1955

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT

INVESTIGATOR: Donald G. Coleman

CONTRACT: DO 33(616)53-20

CONTRACTOR: Forest Products Laboratory

ABSTRACT: Developments in the program of research in sandwich construction for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1955 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Eight technical reports issued during the fiscal year are abstracted.

WADC TR 55-463

March 1956

SUBJECT: DEVELOPMENT OF BRAZED SANDWICH CONSTRUCTION MATERIAL FOR HIGH-TEMPERATURE APPLICATION

INVESTIGATOR: W. Maxwell, J. Mueller,  
J. Siltanen

WADC TR 53-373 Sup 3

CONTRACT: AF 33(616)-2602  
CONTRACTOR: The Glenn L. Martin Company  
ABSTRACT: Procedures were developed for brazing honeycomb cores to stainless steel skins to form sandwich material suitable for elevated temperature applications. After a preliminary investigation, 17-7PH stainless steel skin and core material and Coast Metals No. 53 brazing alloy were selected for the process development work. Several brazing methods were studied and muffle brazing in an inert atmosphere was selected for the production of two-by-two foot panels. The protected-core-container methods was also developed and its feasibility demonstrated on a two-by-two foot panel. The large panels produced will be supplied to the Materials Laboratory, Wright Air Development Center, for testing and evaluation. During the course of the investigation, flexure testing and metallographic examinations were used for general screening and evaluation of the methods, heat treatments, and brazing variables.

TESTS, NONDESTRUCTIVE

WADC TR 55-175 Part 1

January 1956

SUBJECT: METEOROLOGICAL AND SOLAR RADIATION DATA  
INVESTIGATOR: Wm. S. Wilson  
CONTRACT: AF 18(600)-40  
CONTRACTOR: University of Alaska  
ABSTRACT: The daily observations of maximum and minimum temperatures, maximum, minimum, and mean of the relative humidity with the approximate time (Alaskan Standard Time) of the occurrence of each maximum and minimum, maximum intensity of solar radiation and total radiation received on a horizontal surfaces and a surface slanted  $45^{\circ}$  to the south are tabulated along with the monthly averages and totals of each of these factors as obtained in connection with the project on the "Exposure of USAF Materials near College, Alaska." The temperature data for a given year are represented on a composite graph. The monthly values of all data are accumulated in separate tables.

WADC TR 55-175 Part 2

January 1956

SUBJECT: METEOROLOGICAL AND SOLAR RADIATION DATA  
INVESTIGATOR: Wm. S. Wilson  
CONTRACT: AF 18(600)-40  
CONTRACTOR: University of Alaska  
ABSTRACT: The daily observations of maximum and minimum temperatures, maximum, minimum, and mean of the relative humidity with the approximate time (Alaska Standard Time) of the occurrence of each maximum and minimum, maximum intensity of solar radiation and total radiation received on a horizontal surface and a surface slanted  $45^{\circ}$  to the south are tabulated along with the monthly averages and totals of each of these factors as obtained in connection with the project on the "Exposure of USAF Materials near College, Alaska." The temperature data in each year are represented on a composite graph. The monthly values of all data are accumulated in separate tables. The period covered is from 1 September 1954 through 31 August 1955.

WADC TR 53-373 Sup 3

SUBJECT: EVALUATION OF TESTERS FOR THE DETERIORATION OF DOPED-FABRIC SURFACES ON AIRCRAFT

INVESTIGATOR: Bruce K. Bain  
Lawrence P. Slivka

CONTRACT: AF 33(600)-27305

CONTRACTOR: Goodyear Aircraft Corporation

ABSTRACT: Three types of portable doped-fabric testers, the Mobile AMA (Hughes) tester, Seyboth fabric tester, and CAA impact tester, are evaluated as methods of determining deterioration of doped-fabric surfaces on aircraft. Correlation of these testers with the Mullen burst tester is made, and the effects of different temperatures and relative humidities on the tester readings are determined. It is found that the Mobile AMA (Hughes) tester is useful as a survey instrument for determining deterioration of doped-fabric surfaces on aircraft. In their present forms, the CAA and Seyboth testers are found to be useful only for weaker fabrics than those specified by Air Force standards.

#### TEXTILES

WADC TR 54-323 Part 1

December 1955

SUBJECT: THE EFFECT OF SURFACE FINISHES ON FRICTION AND FUSION OF PARACHUTE CLOTH AND LINE

INVESTIGATOR: Vasilis Lavrakas  
Adolph Katz

CONTRACT: AF 18(600)-136

CONTRACTOR: Lowell Technological Institute Research Foundation

ABSTRACT: An attempt to find lubricating agents which would minimize or prevent the fusion of nylon parachute materials caused by frictional heat was the main objective of this program. A friction apparatus, based on the belt friction principle, was developed to evaluate lubricating materials. Scoured parachute materials possessed high frictional forces and fused at low speeds and loads, while lubricated materials possessed lower frictional forces and fused at higher speeds and loads.

Many lubricants were examined and ten were classed as highly effective in preventing fusion. In addition, nylon parachute line coated with these best lubricants was further subjected to breaking strength, fungus resistance, and aging tests. No one lubricant was determined to be superior in all respects. It is recommended that a selection be made among several of those lubricants possessing the most desirable attribute, resistance to fusion, and subjecting them to drop tests at the indicated percentage pickup level.

WADC TR 54-323 Part 2

October 1955

SUBJECT: THE EFFECT OF SURFACE FINISHES ON FRICTION AND FUSION OF PARACHUTE CLOTH AND LINE

INVESTIGATOR: Vasilis Lavrakas  
Adolph Katz

CONTRACT: AF 18(600)-136

CONTRACTOR: Lowell Technological Institute Research Foundation

WADC TR 53-373 Sup 3

ABSTRACT: A belt friction apparatus was used to study the fusion of scoured parachute cloth and lubricated cord at high sliding speeds. Data obtained at speeds up to 97 ft/sec when extrapolated to higher speeds (100- 140 ft/sec) indicate that no further significant data would be obtained regardless of the type of agent applied.

The effect of speed on fusion and friction has also been investigated. Primarily, three homologous series of high molecular weight organic compounds were used: high molecular weight fatty acids, alcohols, and monoesters. In their ability to prevent fusion, fatty acids were poorer than fatty alcohols, while fatty alcohols were poorer than fatty monoesters.

As the speed of rubbing increases, the resistance to fusion of lubricated nylon parachute materials decreases rapidly, and at high speeds (75 ft/sec) this resistance falls to alarmingly low levels.

The role of molecular weight in the phenomenon of fusion and lubrication was investigated and found to be an important factor. Other factors are undoubtedly present but were not investigated.

Anionic, cationic, and nonionic lubricants were also studied. As an insufficient number were investigated, no definite conclusions have been formulated.

WADC TR 54-513

September 1955

SUBJECT: EVALUATION OF ANTISTATIC AGENTS ON NYLON PARACHUTE CLOTH  
INVESTIGATION: James W. Sweeney  
CONTRACT: AF 33(616)-458  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: A literature search covering instrumentation, antistatic agents, and theories of static electrification was conducted to provide a foundation for the evaluation of antistatic treated nylon parachute cloth. An appraisal of the techniques employed by other investigators, in the evaluation of the static behavior of materials, justified the utilization of fabric surface resistance as the measurable parameter. Special instrumentation was developed to provide the desired test conditions of -30°F and 10% RH and permit the required range of resistance measurements. While no permanent antistatic agent studied was found to be effective at the low temperature test conditions, specific non-conditions which was comparable to the resistance of untreated nylon fabric at standard test conditions of 70°F and 65% RH.

WADC TR 54-570

August 1955

SUBJECT: THE EFFECT OF FABRIC STRUCTURE ON THE FRICTIONAL FUSION OF PARACHUTE MATERIALS  
INVESTIGATOR: Vasilis Lavrakas  
CONTRACT: AF 18(600)-136  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: The effect of fabric structure, yarn twist, calendering, and fabric weight on the resistance to fusion of parachute cloth has been studied. A belt friction apparatus, used in lubrication investigations, has also been utilized in this study.

WADC TR 53-373 Sup 3



The parameters of fabric structure, yarn twist, calendering, and fabric weight have been found to be significant in the resistance of parachute cloth to friction.

The results of this study are as follows:

1. The best fabric structure was cloth made according to MIL-C-7020, Type I.
2. Calendered cloth was superior to non-calendered.
3. Low yarn twist and fabric weight appeared to impart higher resistance to fusion than higher twist and weight.

WADC TR 54-571

November 1955

SUBJECT: EVALUATION OF FABRIC FINISHES FOR HIGH TEMPERATURE OPERATION OF PARACHUTE RIBBONS  
INVESTIGATOR: Louis C. Block  
CONTRACT: AF 18(600)-136  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: This research program involved the evaluation of the effect of surface finishes on the high temperature properties of nylon parachute ribbons. The work was conducted at the Lowell Technological Institute Research Foundation from March 1952 to December 1954.

The fabrics were exposed to temperatures above the melting point of nylon (480°F) produced by three modes of heat propagation: conduction, convection, and radiation. Instrumentation and testing programs were developed to appraise the results.

The results showed that no single finish satisfactorily retarded the fusion of the nylon ribbon when it was tested by all three methods. The conclusions drawn were that if a technique to change the heat transfer characteristics of nylon at these high temperatures is to be realized, methods other than applying surface finishes should be investigated.

WADC TR 54-572

November 1955

SUBJECT: AERODYNAMIC HEATING OF PARACHUTE RIBBONS  
INVESTIGATOR: Louis C. Block  
CONTRACT: AF 18(600)-136  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: This research program involved the evaluation of wind tunnel tests on parachute ribbons. The work was conducted at the Lowell Technological Institute Research Foundation from March, 1952, to March, 1954.

A theoretical evaluation of the expected temperature rise of these ribbons in high velocity flight was carried out. Wind tunnel tests were subsequently performed on the basic ribbon and ribbons of modified design.

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The results showed that the experimental values of the heat transfer coefficient agreed very well with the theoretical values. Changes in ribbon design and ribbon material did not effect an improvement to the heat transfer characteristics of the basic sample.

WADC TR 54-574

November 1955

SUBJECT: A STUDY OF THE EFFECTS OF FABRIC GEOMETRY VARIABLES ON AIR PERMEABILITY

INVESTIGATOR: William O. Perry

ABSTRACT: A series of specially designed nylon parachute fabrics was selected to represent extremes in cloth construction and to demonstrate fundamentals of fabric geometry.

By means of a somewhat unusual test arrangement it was possible to indicate the relationship of fabric geometry to air permeability at several pressure differentials. Through the process of establishing the ratio of total fabric area to interstice area, data were obtained on yarn widths as they lie in the cloth. These data were obtained on a variety of twist constructions and will provide a knowledge and background of design data for present application and future studies in this area.

WADC TR 54-611

February 1956

SUBJECT: IMPROVEMENT OF COLORFASTNESS PROPERTIES ON UNITED STATES AIR FORCE FABRICS

INVESTIGATOR: Robert J. Peirent, George O. Langlais,  
Roland E. Darby, Jr.

CONTRACT: AF 18(600)-182

CONTRACTOR: Lowell Technological Institute Research Foundation

ABSTRACT: Dye formulae to provide improved colorfastness on the following United States Air Force fabrics were developed. When tested in a preliminary manner on laboratory materials, the formulae displayed the following colorfastness to light in comparison to that of the standards:

<u>Cloth</u>	<u>Shade Number</u>	<u>Colorfastness of Developed Formulae Compared to Standards</u>
Wool, Serge	Blue 84	Markedly superior
Wool, Gabardine	Blue 156	Markedly superior
Wool, Gabardine	Grey 167	Considerably better
Wool, Gabardine	Tan 193	Equal or slightly better
Nylon, Satin	Sage Green 511	Appreciably better
Nylon, Rayon	Blue 157	Equal or slightly better

The formulae developed for Blue 84 and Blue 156 withstand more than 80 hours Fadeometer exposure without producing a visual break in color.

The formula developed for Gray 167 displays only a moderate change in color after 140 hours of Fadeometer exposure --- an exposure that destroys the color of the standard almost completely.

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Tests of the colorfastness of the 100-yard length of each of the first four fabrics listed which were produced using the developed formulae in the dyeing operations substantiated the laboratory-evaluated colorfastness results with one exception:

The color on the Tan 193 length was slightly less fast than the standard.

The last two fabrics listed, i.e., those involving shades Sage Green 511 and Blue 157, were carried through to the development of dye formulae --- no fabric was manufactured.

The development of the dye formulae, the problems encountered in establishing them properly in fabric manufacture, and the test measurements of the properties of the fabrics produced are presented.

WADC TR 55-83

January 1956

SUBJECT: THE DEVELOPMENT OF WOOL-SYNTHETIC BLENDED FABRICS FOR SUMMER FLIGHT GARMENTS  
INVESTIGATOR: George O. Langlais  
Samuel L. Fuller  
CONTRACT: AF 33(600)-25892  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: A study of wool-synthetic blend fabrics was made to develop one suitable for summer flight clothing.

A survey of worsted manufacturers was conducted to obtain commercially available blend fabrics that approximated the basic requirements. The results of tests performed on the fabrics obtained were considered in subsequent design and manufacture.

Identically constructed fabrics composed of blends of nylon, Dacron, Orlon, and Dynel (each blended at 15%, 30%, and 60%) with wool and an all-wool control were produced; these were tested and compared with respect to fabric properties.

Particular emphasis on the flame-resistance characteristics, specifically centered on the degree of melting and "dripping" after ignition - indicated of major consequence in the establishment of this program - led to the selection of the final fabric, a 70% wool-30% Dynel fabric.

The unfavorable results obtained in this program on any of the synthetic fiber-wool blends and constructions, is not to be construed as an indication of the performance of other blends or constructions or where other parameters of tests and performance are stipulated.

WADC TR 55-104

February 1956

SUBJECT: STUDY OF THE EFFECT OF TWIST IN YARNS ON PARACHUTE FABRICS  
INVESTIGATOR: Chauncey C. Chu, Milton M. Platt,  
Charles A. Lermond

WADC TR 53-373 Sup 3

CONTRACT: AF 33(616)-387

CONTRACTOR: Fabric Research Laboratories, Inc.

ABSTRACT: This is the final report on the "Study of the Effect of Twist in Yarns on Parachute Fabrics." Analytical developments on the mechanics of air flow through textile structures were made by adopting classical flow equations with due consideration to the visco-elastic behavior of textile materials. Experimental results on a large number of fabric samples (MIL-C-7020, Types I and II) with yarn twists varying from 0.5 to 35 turns per inch are given to demonstrate the various changes in the performance characteristics affected by changes in yarn geometry.

From the work accomplished, it is concluded that:

1. The flow of air through the open areas of a fabric obeys the general rules of fluid mechanics namely: flow at any given pressure differential varies with the amount of open area; and the rate of flow at varying pressure differentials follows the square root of the pressure differential with suitable modifying constants to allow for those changes in the open area which occur when the fabric is subjected to biaxial extensions exerted by the air pressure.
2. The free area available for air flow varies as a function of fabric and yarn geometry. The ellipticity of the yarn cross section is functional with the yarn twist; flatter the yarn, the less is the open space between adjacent yarns. Hence, for a given texture (threads per inch) the free area varies inversely with the yarn width.
3. The open areas change when the fabric under test is subjected to increased pressure differentials. The yarn systems in the fabric structure, when so stressed, result in biaxial extensions which widen the spaces between yarns. The rate at which the open areas vary with pressure differential may be determined by studying the fabrics' biaxial stress-strain behavior. At the present writing only limited studies have been made.
4. The magnitude of open areas in any given fabric may be calculated from the yarn widths determined microscopically and the fabric texture. However, a more precise method has been developed by measuring light penetrability through the use of a Beckman Spectrophotometer.
5. In general, the following trends have been shown to be evident: with the increase of yarn twist

Fabric thickness	increased
Denier of yarn removed from fabric	increased
Horizontal yarn diameter	decreased
Vertical yarn diameter	increased
Free area: area between yarns	increased
Light penetrability	increased
Air permeability	increased
Freedom of yarn slippage	increased
Yarn tensile strength	increased
Yarn elongation	increased
Energy to rupture yarn	increased
Strip tensile strength	no visible change
Fabric elongation	no visible change
Secondary creep	increased
Tear strength	decreased
Tear energy	decreased

WADC TR 55-264

February 1956

SUBJECT: HANDBOOK OF PARACHUTE TEXTILE MATERIALS AND PROPERTIES  
 INVESTIGATOR: James W. McCartv  
 CONTRACT: AF 33(616)-2473  
 CONTRACTOR: Georgia Institute of Technology  
 ABSTRACT: This report contains the summarized and consolidated information extracted from several WADC Technical Reports covering several phases of parachute-textile research. The information is arranged to make the results of these reports more readily available and more useful to parachute designers and others interested in the parachute-textile phase of Air Force work.

The report is divided into sections covering the pertinent facets of parachute-textile information. Fairly complete information is supplied on elongation, elastic recovery, energy absorption, porosity and air permeability, strength properties, and temperature properties of numerous different textile yarns, cords, and fabrics. Some information is also supplied on aging properties, basic design data, and impact loading.

WADC TR 55-324

January 1956

SUBJECT: DEVELOPMENT OF CHEMICALLY RESISTANT, HIGH-TEMPERATURE PROTECTIVE FABRIC  
 INVESTIGATOR: A. S. Kidwell  
 CONTRACT: AF 33(616)-2544  
 CONTRACTOR: The Connecticut Hard Rubber Company  
 ABSTRACT: A coated fabric comprising a thin coating of aluminum-pigmented polychlorotrifluoroethylene on glass fabric backed with a low-density silicone rubber sponge, and having an overall thickness of 0.070 inch, was developed for use in protective clothing for personnel in danger of being exposed briefly to a chemical fire at temperatures up to 1000°F. The fabric shows excellent resistance to fuming nitric acid and other oxidizers and fuels, is flexible and useful over a temperature range of -80°F to +390°F.

WADC TR 53-373 Sup 3

This fabric construction showed a temperature rise on the inside surface of less than 100°F after direct exposure to a flame caused by mixing white fuming nitric acid and monoethylaniline. Peak-flame temperatures averaging about 1600°F three seconds after mixing the reactants were measured with thermo-couples. The fabric was non-porous to 1000°F steam, and was not penetrated by reaction products of the ethyl aniline-fuming nitric acid combustion.

Polyethylene and polyethylene-Vistanex blends were cured with peroxide and were resistant to temperatures of 250°F. They also showed improved resistance to penetration by white fuming nitric acid.

WADC TR 54-573

November 1955

SUBJECT: DESIGN AND DEVELOPMENT OF A HEAVY WEIGHT HIGH IMPACT SHOCK MACHINE  
INVESTIGATOR: Edward N. Sabbagh, Arthur S. Quint  
CONTRACT: AF 18(600)-127  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: The construction and operation of a pneumatically driven impact tester designed to evaluate the dynamic performance characteristics of cushioning materials is described. The 577-pound flat impacting element is capable of being projected downward with a velocity of from 20 ft/second to over 50 ft/second. An analog computer is used in a new application to record and analyze the impact.

WADC TR 55-229

February 1956

SUBJECT: PERFORMANCE CHARACTERISTICS OF CUSHIONING MATERIALS IMPACTED UNDER A HEAVY WEIGHT HIGH IMPACT SHOCK MACHINE  
INVESTIGATOR: E. N. Sabbagh  
CONTRACT: AF 18(600)-127  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: The energy absorption characteristics of cushioning materials impacted under a heavy weight high impact shock machine and analyzed by an analog computer system are reported. The dynamic performance curves in the form of graphs of energy versus maximum stress and maximum stress versus maximum strain are given for a wide variety of materials.

WADC TR 55-343

January 1956

SUBJECT: PERFORMANCE CHARACTERISTICS OF PAPER HONEYCOMB CUSHIONING MATERIALS IMPACTED UNDER A HEAVY WEIGHT HIGH IMPACT SHOCK MACHINE  
INVESTIGATOR: Edward N. Sabbagh  
CONTRACT: AF 18(600)-127  
CONTRACTOR: Lowell Technological Institute Research Foundation  
ABSTRACT: Dynamic tests with respect to the energy absorption characteristics of certain paperboard honeycomb materials impacted under a heavy weight high impact shock machine show that these materials are more efficient energy absorbers at the lower limit of the imposed test conditions than other materials previously tested. They are much less effective in the higher portions of the test range.

A correlation between energy absorption and density is exhibited.

February 1956

SUBJECT: DEVELOPMENT OF DACRON PARACHUTE MATERIALS  
INVESTIGATOR: Hamilton J. Bickford, Donald K. Kuehl ,  
Thomas L. Rusk, Jr.  
CONTRACT: AF 33(616)-2562  
CONTRACTOR: Cheney Brothers  
ABSTRACT: Dacron fabrics can provide excellent dimensional stability up to 350°F and possibly 400°F.

Dacron parachute fabrics do not lose appreciable strength on exposures at 350°F up to 16 hours.

Losses in strength will occur on long exposures at 400°F.

Proper design will allow the attainment of almost any air permeability range (with reasonable tolerances) for Dacron fabrics ranging from 2 ozs/sq yd up to at least 14 ozs/sq yd. The air permeability range of fabrics weighing over 14 ozs/sq yd is limited by the amount of sleaziness acceptable in the fabric. Permeabilities over 50 cu ft/min/sq ft at 1/2-inch pressure differential can be achieved, but only by using an open weave that distorts too readily to be practical.

Seam efficiencies of 90% cannot consistently be obtained on Dacron fabrics with the present knowledge of seam performance.

Dacron can be processed to provide a balanced fabric (coinciding load-elongation curves within 15%).

WADC TR 56-15

February 1956

SUBJECT: THE EFFECTS OF GAMMA RADIATION ON TEXTILE MATERIALS  
INVESTIGATOR: Joyce McGrath  
R. H. Johnson  
ABSTRACT: The primary purpose of this investigation was to obtain data on the effect that exposure to high energy gamma radiation, for specific periods of time, would have on various parachute textile materials, also to determine if the use of parachute marking ink would increase or speed up the degradation of the materials.

The materials were irradiated both in the Cobalt-60 source located in Materials Laboratory, Wright Air Development Center, WPAFB, Ohio, and the Materials Test Reactor, National Reactor Testing Station, Idaho Falls, Idaho. All materials were evaluated both before and after exposure.

The white nylon fabrics showed the greatest loss in strength after exposure for sixteen hours at a total dosage of  $7.4 \times 10^6$ r, (over 25% loss), while the Dacron\* fabrics retained a high percentage of strength for the same exposure (less than 7% loss). The loss in strength of both nylon and Dacron

\*E. I. du Pont de Nemours's fiber

fabrics marked with parachute marking ink, was no greater than for the same fabrics that were not marked with the ink.

All webbings and cords had a high percentage of loss after exposure for 18.3 hours with a total dosage of  $1.7 \times 10^8$ r. In this group the nylon webbing, Spec. Mil-W-4088, Type XIII, resin treated gave the best results, with a loss of 26-28%. All types of cord showed a loss in strength of 45 to 53%.

Nylon fabric from parachutes which were exposed to varying degrees of thermal and nuclear radiation in tests at the Nevada Proving Grounds showed very slight degradation except in areas where identification nomenclature had been stamped. (Spec. Mil-I-6903 Ink, Marking, Blue). In several instances the identification area was burned or melted out in the outline of the identifying cipher or letter. In others the area was so completely damaged that identification was impossible.

### TRANSPARENT MATERIALS

WADC TR 54-128 Part 2

October 1955

SUBJECT: EVALUATION OF MATERIALS FOR GUNSIGHTING DOMES Pt. 2 Long  
Time Tensile Strength Evaluation  
INVESTIGATOR: Charles R. Frownfelter, Duane C. Shearer  
CONTRACT: AF 33(616)-106  
CONTRACTOR: Goodyear Aircraft Corporation  
ABSTRACT: This report covers a series of long-time tensile tests on laminated transparent plastic materials suitable for forming gunsighting domes. These tests were part of an evaluation which also included (Reference Part 1 of this report) adhesive tests, heat stability tests, and ultimate tensile tests at various temperatures. The materials investigated were regular grade laminated acrylic (BMS8-4), heat resistant grade laminated acrylic (MIL-P-7524), and modified acrylic face sheets (5105XP) laminated with a standard polyvinyl butyral interlayer. The 5105XP laminate was supplied by two laminators.

The long-time tensile tests consisted of subjecting specimens of the above laminated materials to constant stresses of (nominally) 4000, 2000, 1000, and 750 psi for periods up to 16 months under two conditions: (1) outdoor exposure in South Florida, and (2) indoor room temperature exposure at the Goodyear Aircraft plant in Akron.

From the standpoint of time to fail at a given stress, laminated 5105XP was the best of the materials by a wide margin, having withstood 1000 psi for at least 11,000 hours in Florida and about 2000 hours at room conditions. Natural weathering in Florida also promoted delamination between the face sheets and the butyral interlayer, particularly in the laminated 5105XP material supplied by one of the two laminators, and in the BMS 8-4 material.



SUBJECT: STABILIZATION OF POLYMETHYL ALPHA CHLOROACRYLATE PLASTIC SHEET

INVESTIGATOR: Harry D. Anspon

CONTRACT: AF 33(600)-23883

CONTRACTOR: General Aniline and Film Corporation

ABSTRACT: When this contract was initiated, it already had been shown that polymethyl  $\alpha$ -chloroacrylate plastic sheet had outstanding physical properties - high heat distortion temperature, high tensile and flexural strengths, excellent craze resistance, low notch sensitivity, unique self-extinguishing property in burning tests, and complete formability - which made it particularly suited for use as an aircraft glazing material especially if its heat and light stability could be improved.

In order to obtain this plastic as a completely acceptable aircraft glazing material, Wright Air Development Center awarded this contract for the improvement of the heat and light stability of polymethyl  $\alpha$ -chloroacrylate plastic sheets.

As a result of the work completed under this contract, polymethyl  $\alpha$ -chloroacrylate plastic sheets possessing outstanding heat and light stability were prepared and were submitted to Wright Air Development Center.

This report summarizes the research and development work involved in obtaining these heat and light stabilized polymethyl  $\alpha$ -chloroacrylate plastic sheets.

The detailed outstanding properties of polymethyl  $\alpha$ -chloroacrylate (PMACA) established by testing programs on previous samples of polymethyl  $\alpha$ -chloroacrylate plastic sheets are summarized below:

1. PMACA possesses a heat distortion temperature of 130-140°C (266-284°F).
2. PMACA possesses a tensile strength of 17,000 psi at room temperature and even at 110°C (230°F) it possesses a tensile strength of 8,000 psi.
3. PMACA possesses a flexural strength of 23,000 psi at room temperature.
4. Even though PMACA is quite hard (Barcol Hardness of 65), it still exhibits excellent strength properties at room temperature after notching; for its notched tensile strength is 12,000 psi and its notched flexural strength is 13,000 psi.
5. PMACA exhibits good resistance to solvent and stress crazing. PMACA samples withstood 11,000 psi flexural stress for 23 hours before crazing and 5,000 hours before failing

in a long time flexural test. The also withstood 9,000 psi stress under toluene, 8,000 psi stress under isopropyl alcohol, and 4,500 psi stress under methyl ethyl ketone, for over 300 seconds without crazing.

6. PMACA is completely formable. At 177°C (350°F) a 6 in. diameter hemispherical dome was blown from 1/4 in. thick sheet with 18 psi air pressure.
7. PMACA is rapidly self-extinguishing. When tested for flammability by Method 2021 of Federal Specification L-P-406a, the flame extinguished immediately after removal of the external burner flame.

Stabilization of PMACA plastic sheets against heat and light is believed to overcome the principal deterrent to the use of PMACA, with its superior properties as an aircraft glazing material.

WADC TR 55-412

December 1955

SUBJECT: ELEVATED AND ROOM TEMPERATURE PROPERTIES OF SIERRACIN 611, PMACA, AND PLEXIGLAS 55 TRANSPARENT PLASTIC SHEET MATERIALS

INVESTIGATOR: John A. Vanecho, Ward F. Simmons, Gale R. Remely

CONTRACT: AF 33(038)-10818

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Creep and creep-rupture, short-time tensile, and deterioration properties of two transparent plastic sheet materials, Sierracin 611, a polyester, and PMACA, an acrylate, were determined over a temperature range of 80 to 300 F. Some creep and creep-rupture and tensile data were also obtained on Plexiglas 55, an acrylic transparent, for comparison with the strength properties of an experimental acrylic, 5105XP.

The PMACA sheet material displays, in general, at least 50 per cent greater creep, creep-rupture, and short-time tensile strength and elastic-modulus properties than do Sierracin 611 and Plexiglas 55. This PMACA material compares almost equally as favorable with other transparent plastics previously tested under this evaluation program, such as acrylates Polymer K, 5105XP, Lucite HC-202, and Plexiglas II and the polyester resin Selectron 44.

The deterioration losses determined for PMACA and Sierracin 611 remained relatively low for temperatures as high as 250 F. Of the three transparents tested, crazing was shown to occur in the PMACA and Plexiglas 55 materials.

WADC TR 55-217

January 1956

SUBJECT:

INVESTIGATOR: Philip Chen, Stephen Giacoppe, B. David Halpern, Wolf Karo, Philip Levine, C. Leroy Tibery

CONTRACT: AF 33(600)-26409

CONTRACTOR: Monomer-Polymer

WADC TR 53-373 Sup 3

**ABSTRACT:** The objective of the project was the development of a safety glass interlayer material which would withstand temperatures up to 500°F. Copolymers of ethyl acrylate and acrylonitrile were studied first. Polyacrylates alone were found to be more stable than the copolymers of the acrylates with acrylonitrile.

Since the preparation of a laminate to test the heat stability of a polymer proved cumbersome, small samples of the polymers to be tested were prepared in test tubes. A number of acrylates including the acrylates of fluorinated alcohols such as trifluoroethanol, and copolymers of these acrylates with acrylonitrile, methacrylonitrile, and several acrylamides were evaluated by test tube tests.

None of the polymers tested appeared capable of withstanding the elevated temperatures specified. The homopolymers of the acrylates were found to withstand high temperatures better than the copolymers with other monomers such as the acrylamides.

To determine the structural requirements of acrylate esters for the greatest resistance to thermal degradation, a series of polyacrylates was decomposed at 360°C. under vacuum, and the decomposition products were isolated and investigated.

Analysis of the mechanism of thermal degradation of acrylates based on identification of some of the decomposition products indicated that the alpha hydrogen of the acid fragment and the hydrogens on carbon atoms immediately adjacent to the carbonyl carbon of the alcohol moiety were associated with the instability of the polymer. When monomers were synthesized in which these critical hydrogens were not present, polymers could be prepared which were significantly more resistant to thermal degradation than the more commonly known acrylates.

WADC TR 55-272

January 1956

**SUBJECT:** DEVELOPMENT OF ELECTRICALLY CONDUCTIVE TRANSPARENT COATINGS FOR ACRYLIC PLASTIC

**INVESTIGATOR:** Myron A. Coler, Arnold S. Louis, John C. Kalbach

**CONTRACT:** AF 33(616)-2027

**CONTRACTOR:** Markite Company

**ABSTRACT:** The Marklad process, a procedure involving repeated contact between a plastic surface being treated and small particles previously coated with a transferable conductor, has been adapted to the application of transparent static dissipating coatings to acrylic plastic, including curved surfaces such as occur in aircraft canopies.

It has been established that coated meter windows having resistivities readily attained by the Marklad process will adequately protect sensitive meter movements against external static charges or the accumulation of charges on the windows themselves.

WADC TR 53-373 Sup 3

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Flat sheets have been coated so as to have resistance in the range of 1 to 10 megohms per square, light transmission of 75 to 85%, and haze of 1.0 to 2.5%. Uniformity has been improved so that 90% of the area of a flat sheet is encompassed within one order of magnitude. Methods are indicated for having all areas within these limits. Procedures have been extended in the direction of handling curved surfaces. Undercoats and overcoats which promote the uniformity of Marklad coatings and protect them against wear and washing have been developed. Undercoated and overcoated antistatic coatings have withstood the tests of Fed. Spec. L-P-406b, Method 1093, Spec. MIL-C-5547 and Spec. MIL-P-80A without deterioration of anti-static properties.

WADC TR 55-296

December 1955

SUBJECT: RESEARCH, DEVELOPMENT, AND FABRICATION OF SILOXANE-ACRYLATE INTERLAYER MATERIAL

INVESTIGATOR: B. David Halpern, Leroy Tibery, Wolf Karo,  
William Prosk, John Isler, Jr.

CONTRACT: AF 33(616)-2528

CONTRACTOR: Monomer-Polymer

ABSTRACT: The object of this research, development, and fabrication program was for the production of interlayer sheet material. This interlayer material consisting of a copolymer of 99 parts of ethyl acrylate and one part of vinyl polysiloxane was developed under Air Force Contract 33(600)-22723 and is described in WADC TR 54-57

This developmental work was undertaken between 15 June 1954 and 15 June 1955

Attempts to mill and calender the interlayer material were unsuccessful. This necessitated the production of interlayer sheets by casting between glass plates. Thermal polymerization was substituted for ultraviolet polymerization for quality production purposes. Sheets produced by this method, however, were optically imperfect.

An adhesive was required for laminating the interlayer sheet to glass or plastic. Residual traces of the parting agent, however, prevented good lamination.

Seven 0.200" by 37" by 42" interlayer sheets varying in quality were submitted.

Recommendations are given for methods of improving the optical quality of future interlayer sheets.

WADC TR 54-619 Part 1

July 1955

SUBJECT: THE INVESTIGATION OF MULTIAXIALLY STRETCHED ACRYLIC PLASTIC

INVESTIGATORS: Julius J. Gouza  
Dee A. Hurst

WADC TR 53-373 Sup 3

CONTRACT: AF 33(616)-489

CONTRACTOR: Rohm and Haas Company

ABSTRACT: The effects of stretching acrylic plastic sheet materials were determined so as to indicate the potential value of such procedures for producing transparent aircraft enclosures with properties equal to or better than laminated transparent plastic enclosures.

Specifications MIL-P-8184, 6886A, and 5425A types of acrylic plastic sheet materials have been stretched by two different methods, biaxial and multiaxial, and aged under four different conditions. A biaxial stretching machine was designed and built. Initial material thickness was chosen so as to give a final thickness of approximately 0.25 inch after 0, 25, 50, 75, and 100 percent stretch at temperatures between 248° and 284°F as required for a particular material.

There was no significant difference between the effects of biaxial and multiaxial stretching. Specification MIL-P-8184 type acrylic plastic stretched 75 percent equally in two directions possessed the best properties before and after aging. All stretched materials were better than unstretched in resistance to crazing, impact strength, and notch sensitivity but inferior in resistance to abrasion.

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